

Enantioselective Total Synthesis of (+)-Salvileucalin B

Sergiy Levin, Roger R. Nani, Sarah E. Reisman*

*The Warren and Katharine Schlinger Laboratory for Chemistry and Chemical Engineering, Division of Chemistry and Chemical Engineering, California
Institute of Technology, Pasadena, California 91125
reisman@caltech.edu*

Supporting Information 2 (NMR Spectra):

Sample Name:
RRN-4-229-1
Archive directory:

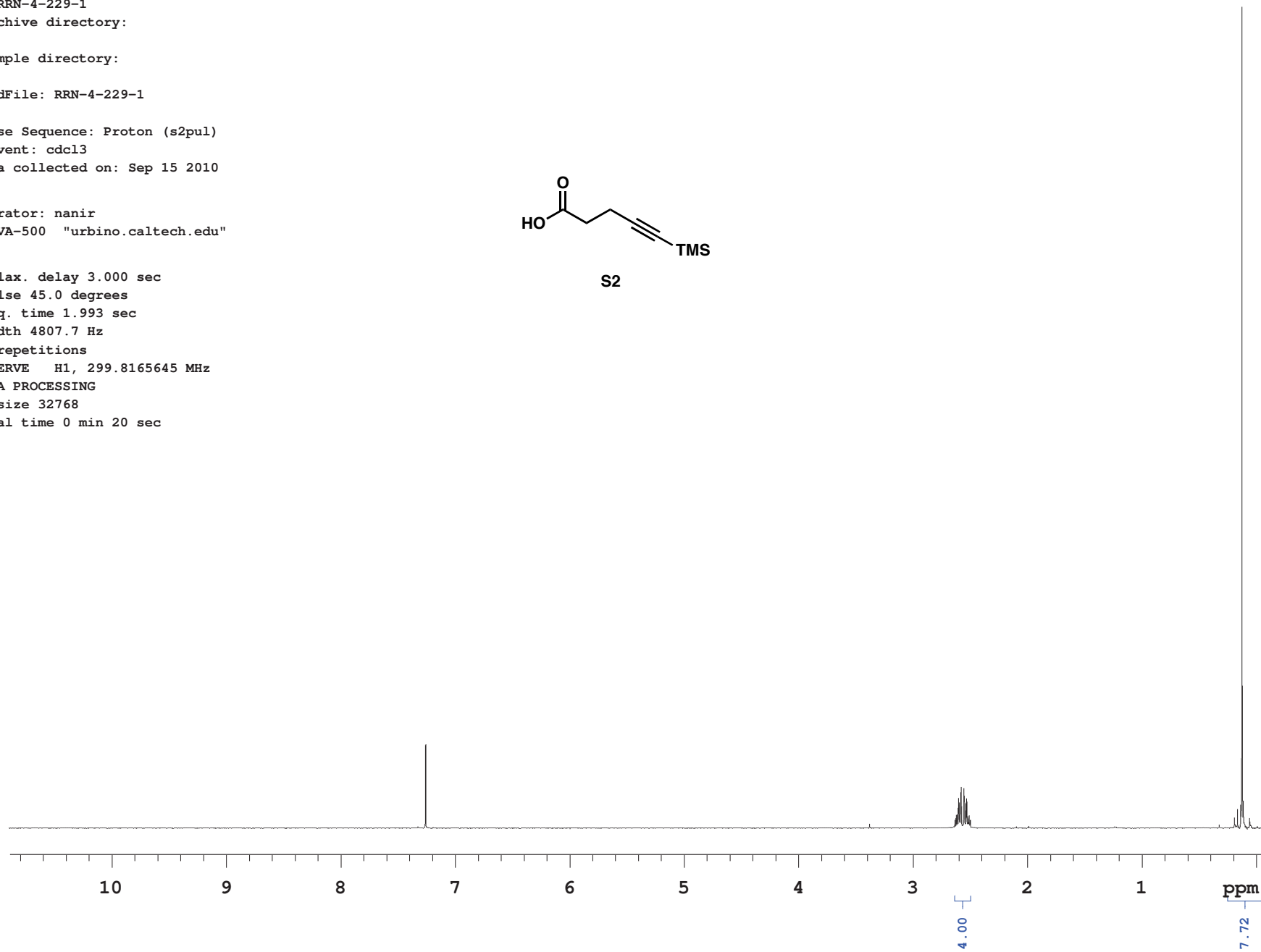
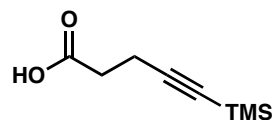
Sample directory:

FidFile: RRN-4-229-1

Pulse Sequence: Proton (s2pul)
Solvent: cdcl3
Data collected on: Sep 15 2010

Operator: nanir
INOVA-500 "urbino.caltech.edu"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.993 sec
Width 4807.7 Hz
4 repetitions
OBSERVE H1, 299.8165645 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 20 sec

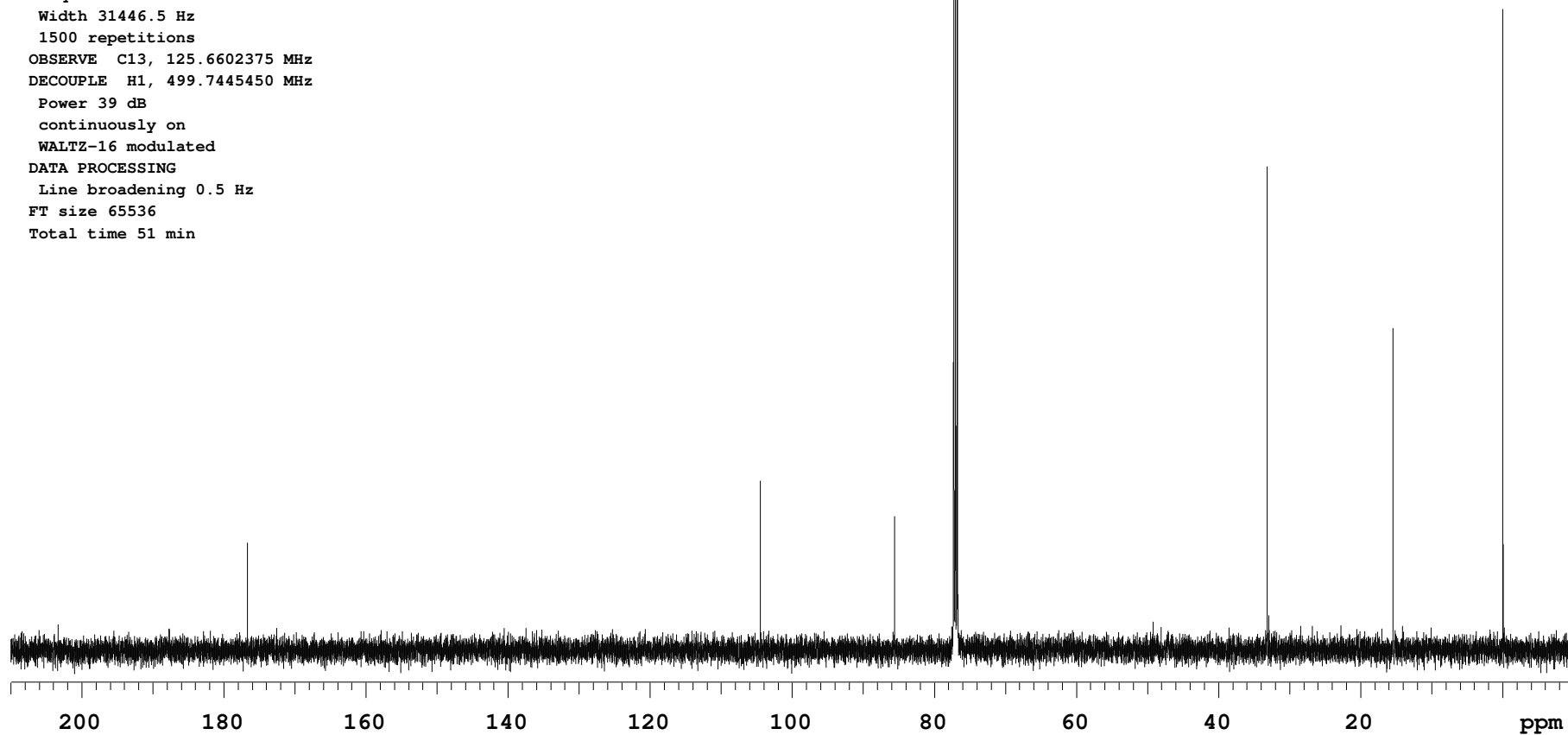
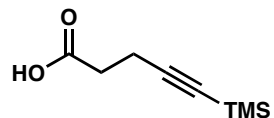


Sample Name:
RRN-4-227-13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-227-13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 15 2010

Temp. 25.0 C / 298.1 K
Sample #39, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602375 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min



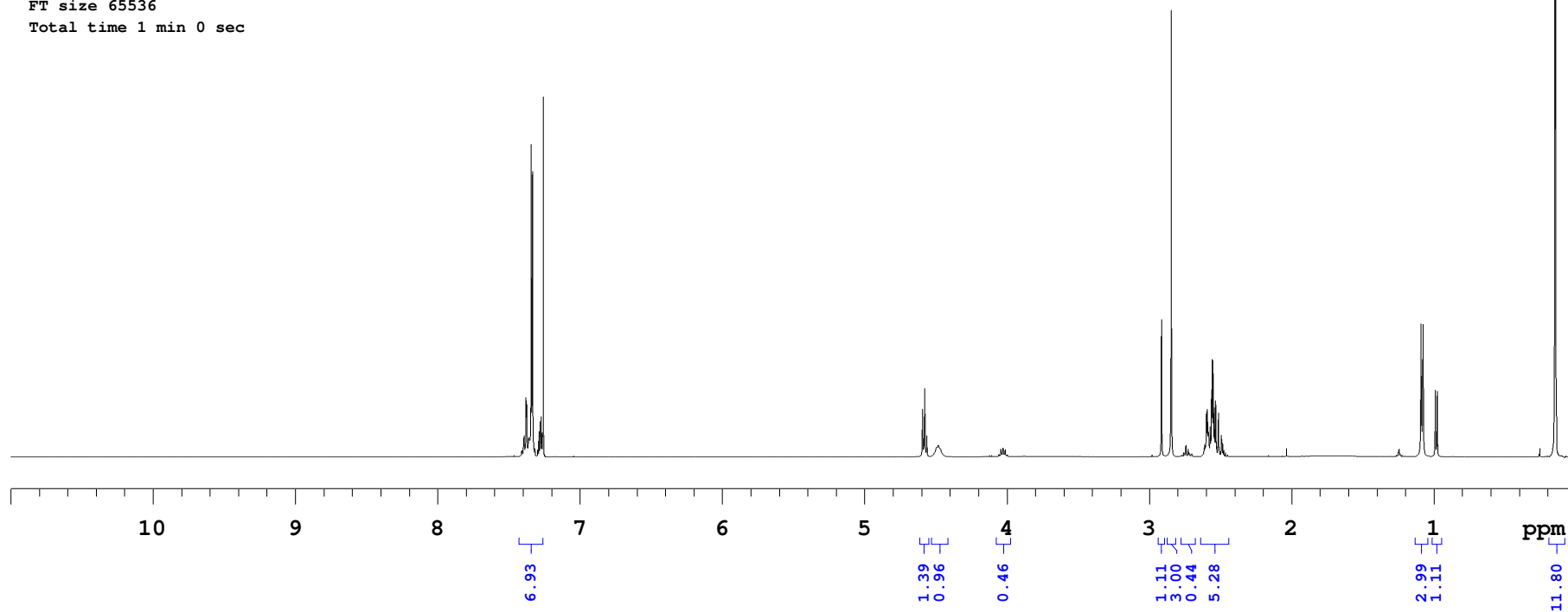
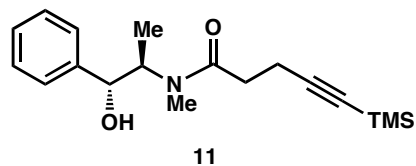
S28

Sample Name:
 RRN-4-231-2
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-4-231-2
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Sep 16 2010

Temp. 25.0 C / 298.1 K
 Sample #44, Operator: nanir

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.7420505 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min 0 sec

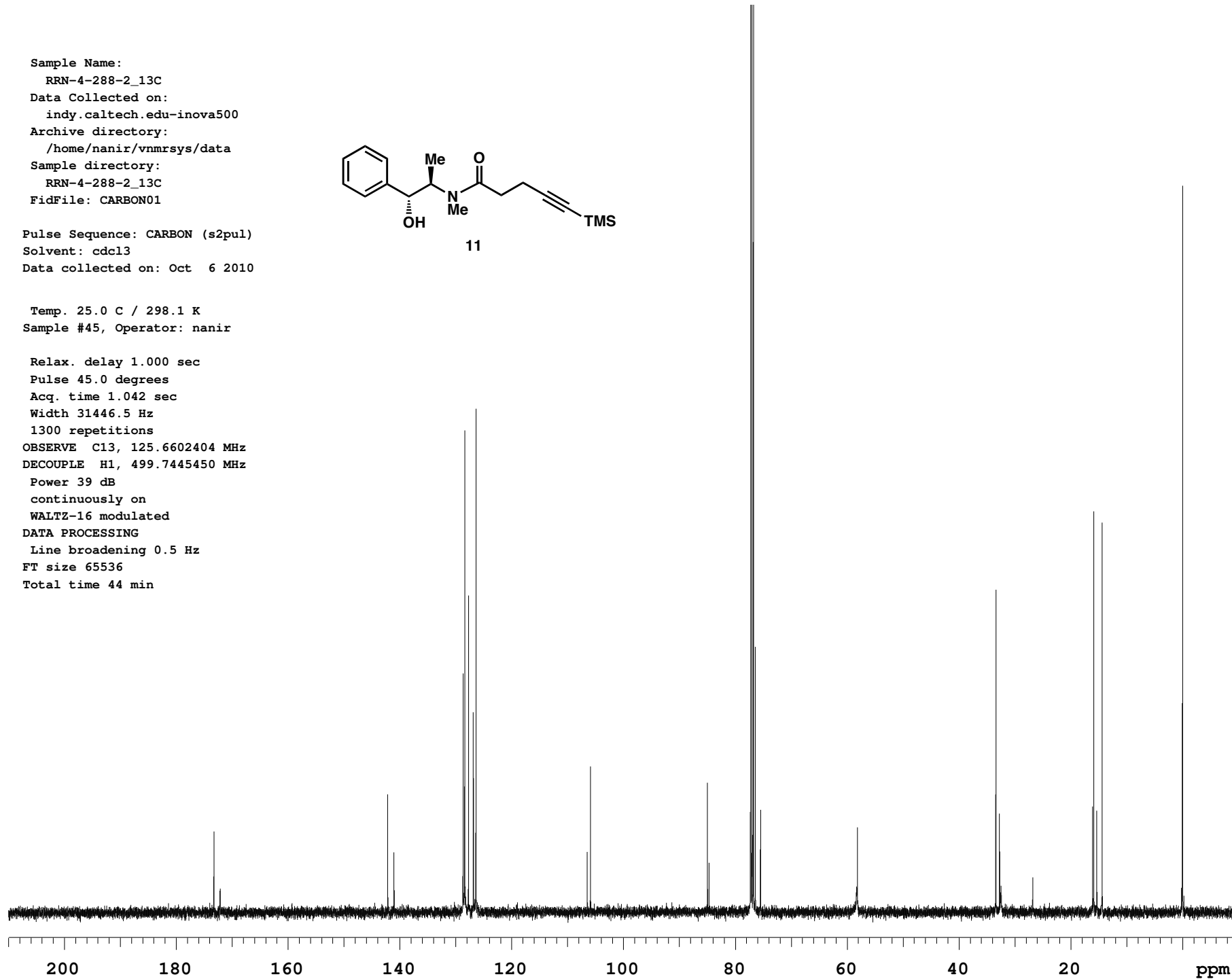
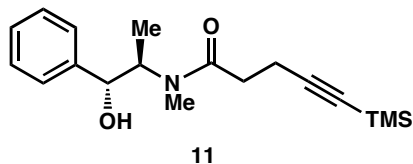


Sample Name:
RRN-4-288-2_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-288-2_13C
FidFile: CARBON01

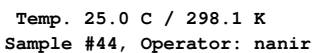
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1300 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 44 min



Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 31 2010



DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 53 sec

10
9
8
7
6
5
4
3
2
1
ppm

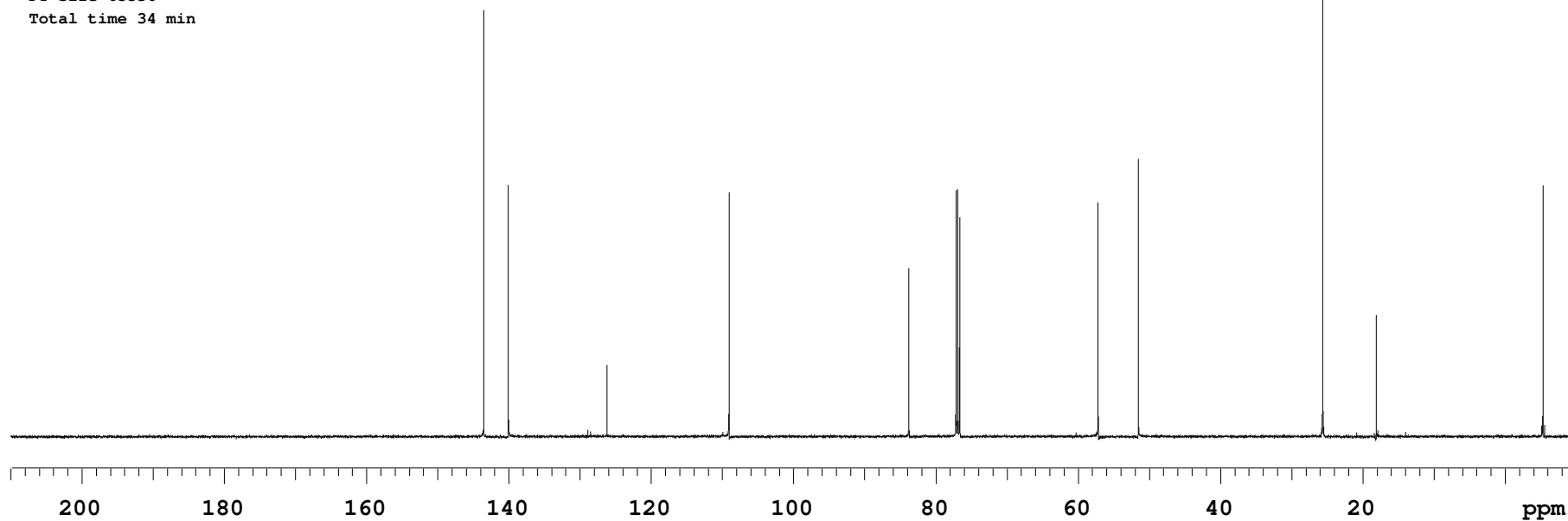
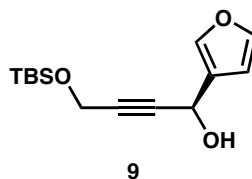
0.97
1.05
1.00
1.00
2.08
9.50
6.06

Sample Name:
RRN-4-180-2_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-180-2_13C
FidFile: CARBON01

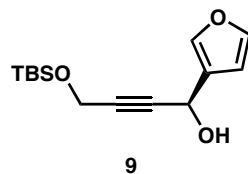
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 31 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

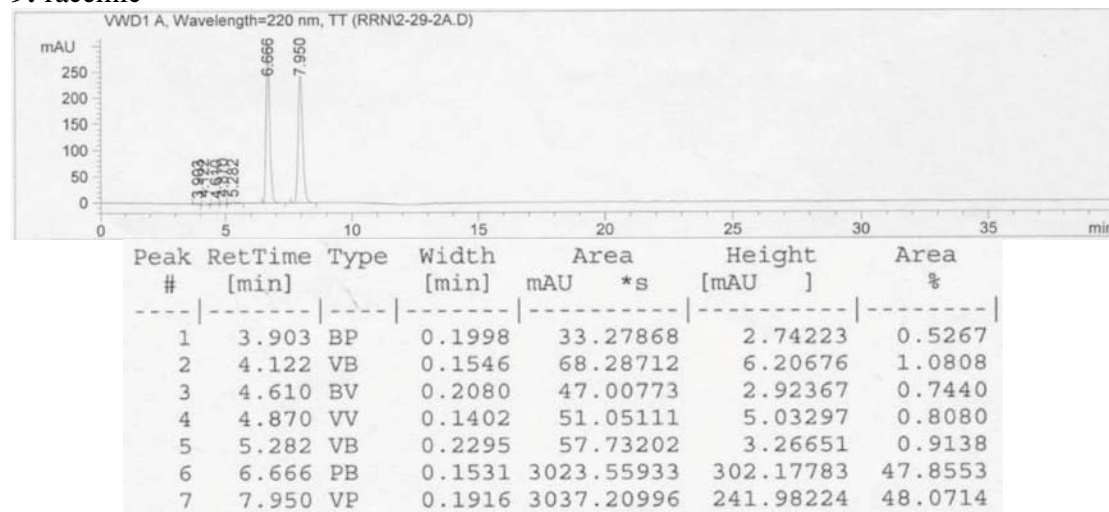
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



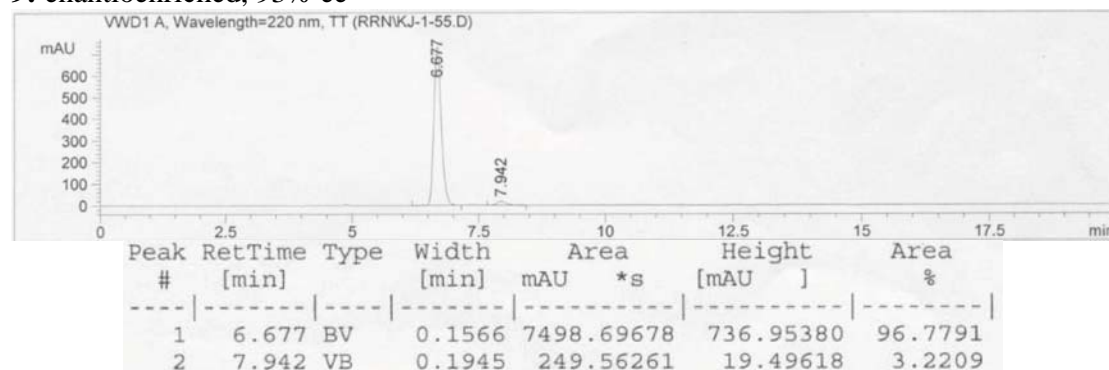
Chromatograms of racemic and enantiomerically enriched propargyl alcohol 9



9: racemic



9: enantioenriched, 93% ee



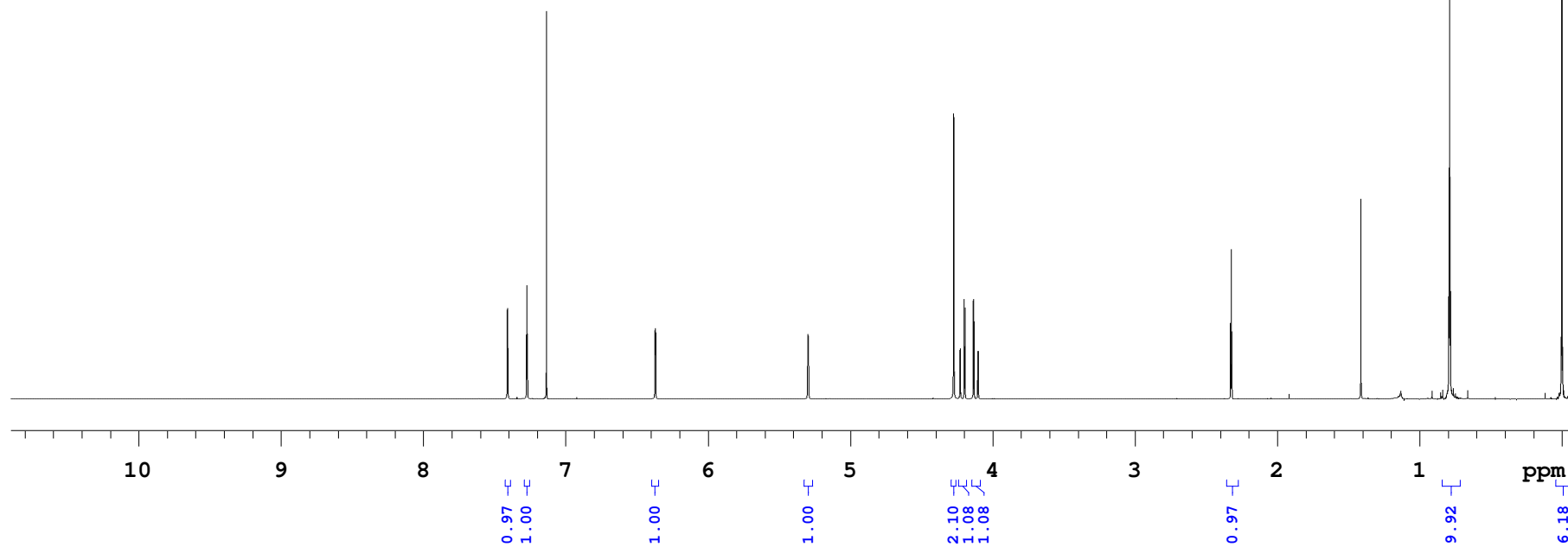
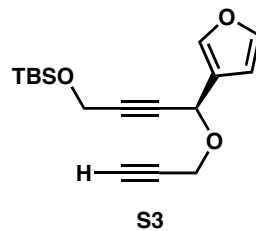
!

Sample Name:
 RRN-4-279-2
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-4-279-2
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
 Sample #45, Operator: nanir

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
 OBSERVE H1, 499.7421115 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 2 min 0 sec



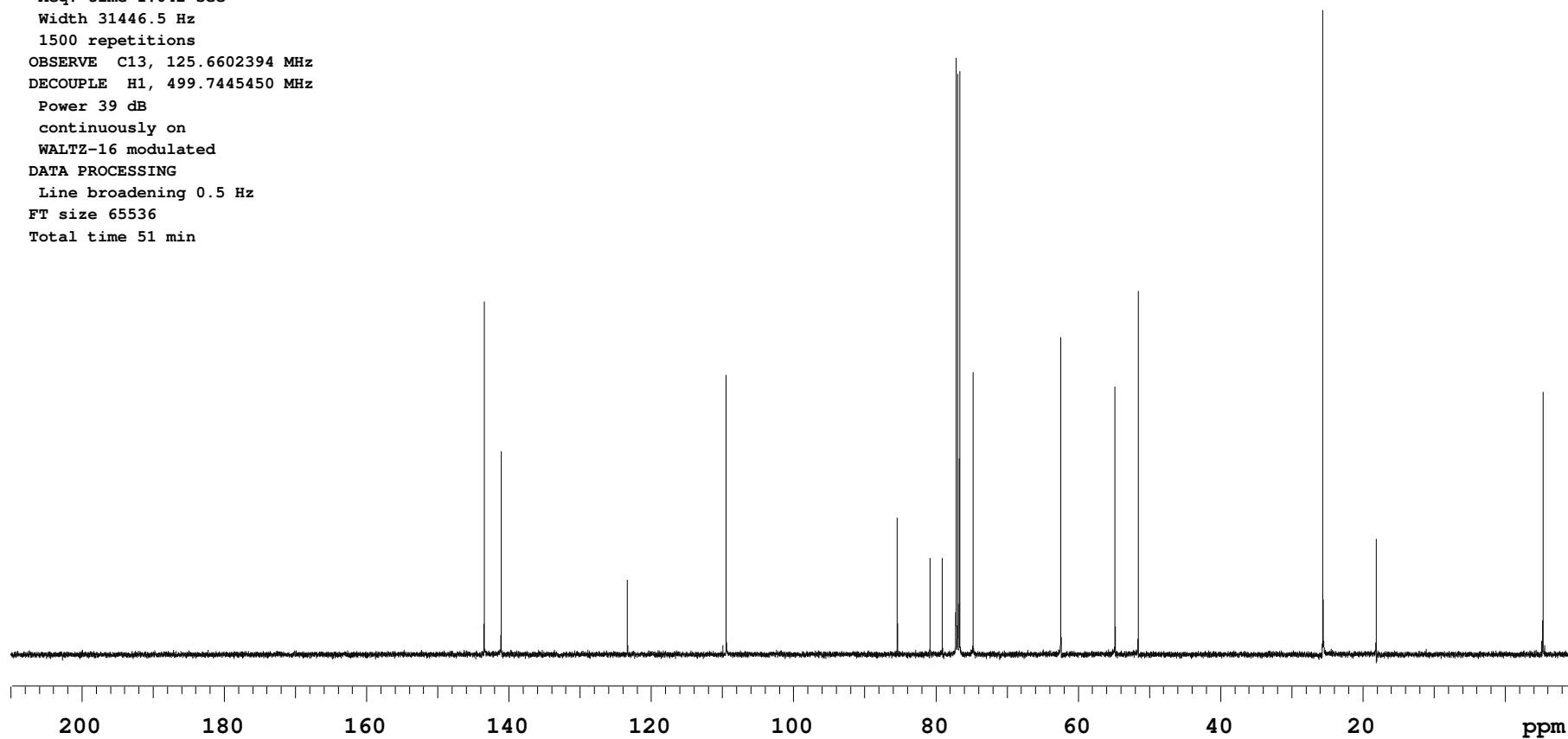
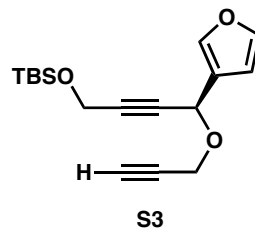
S34

Sample Name:
RRN-4-279-2_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-279-2_13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min



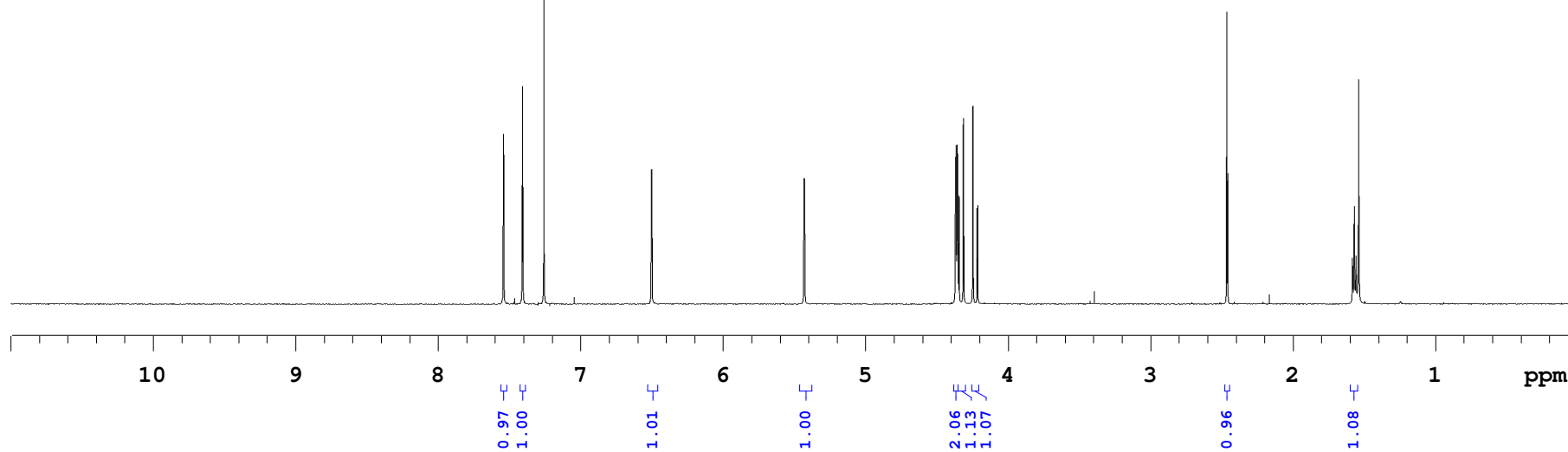
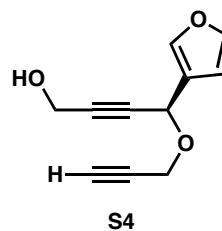
S35

Sample Name:
RRN-4-281-1H_II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-281-1H_II
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010

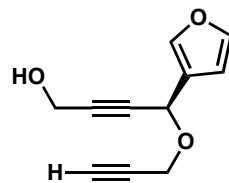
Temp. 25.0 C / 298.1 K
Sample #44, Operator: nanir

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec



Sample Name:
RRN-4-281-13C_II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-281-13C_II
FidFile: CARBON01

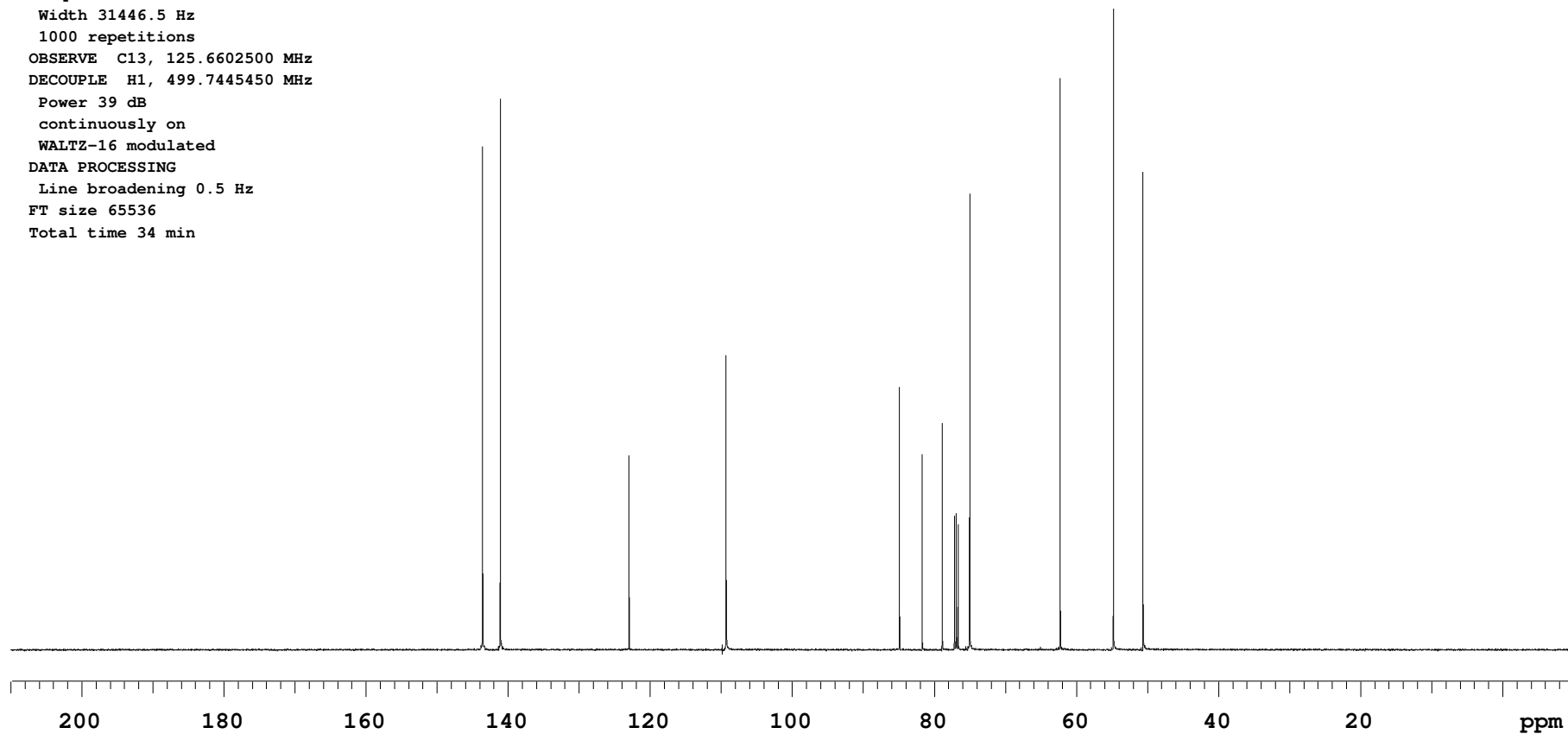
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010



S4

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602500 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



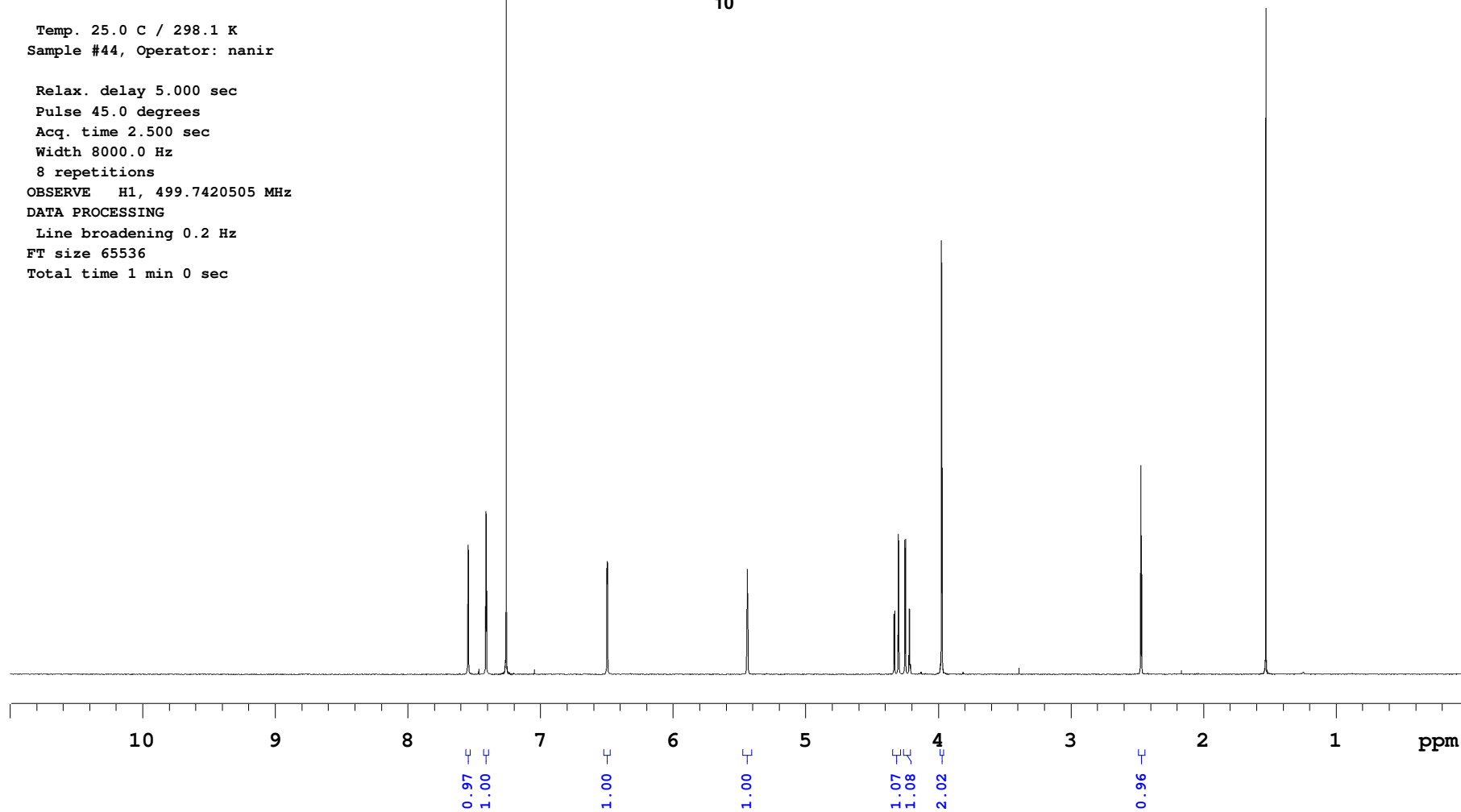
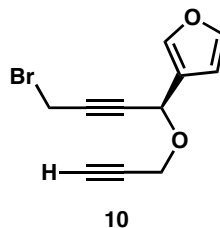
S37

Sample Name:
RRN-4-283-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-283-1
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010

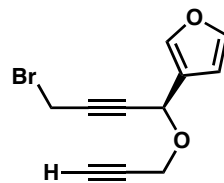
Temp. 25.0 C / 298.1 K
Sample #44, Operator: nanir

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec



Sample Name:
RRN-4-283-1_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-283-1_13C
FidFile: CARBON01

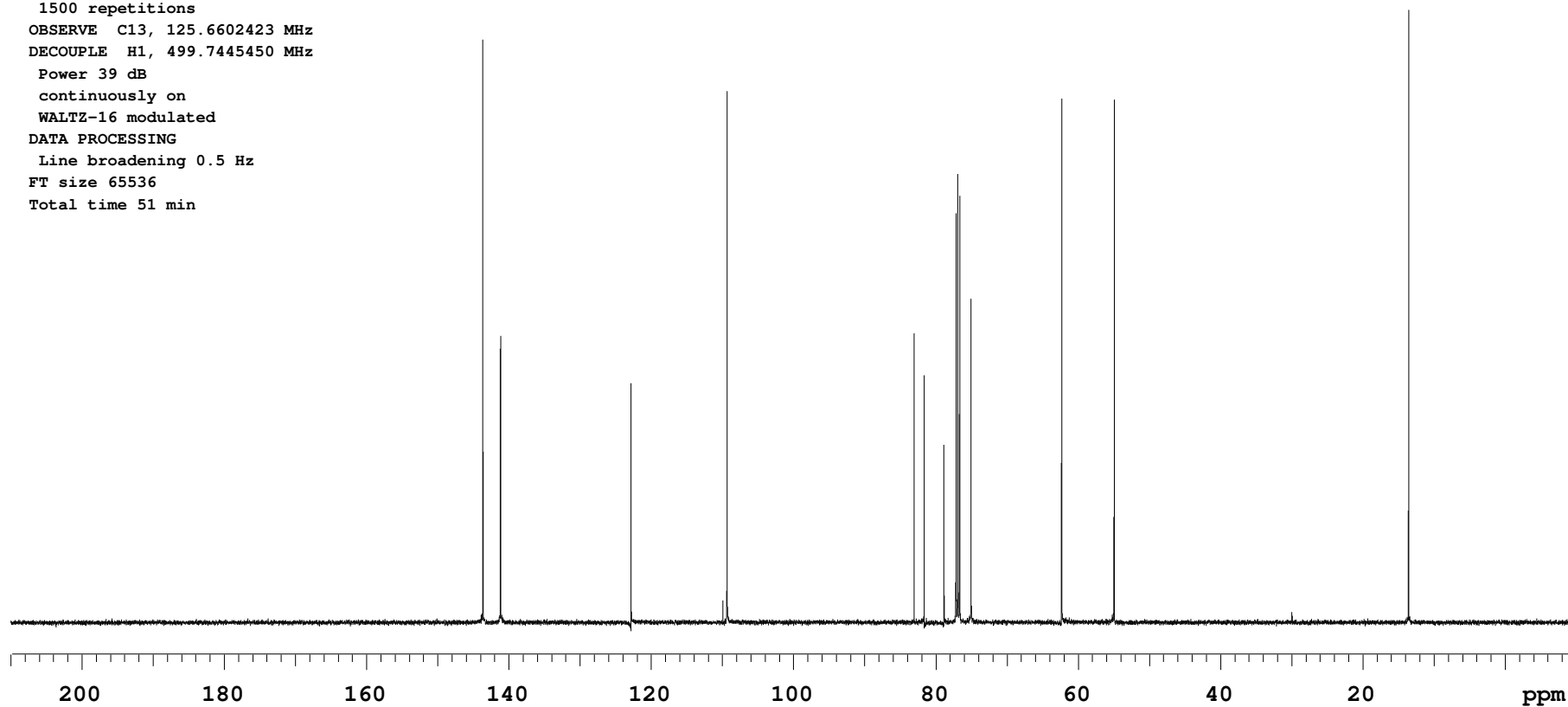
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010



10

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602423 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min

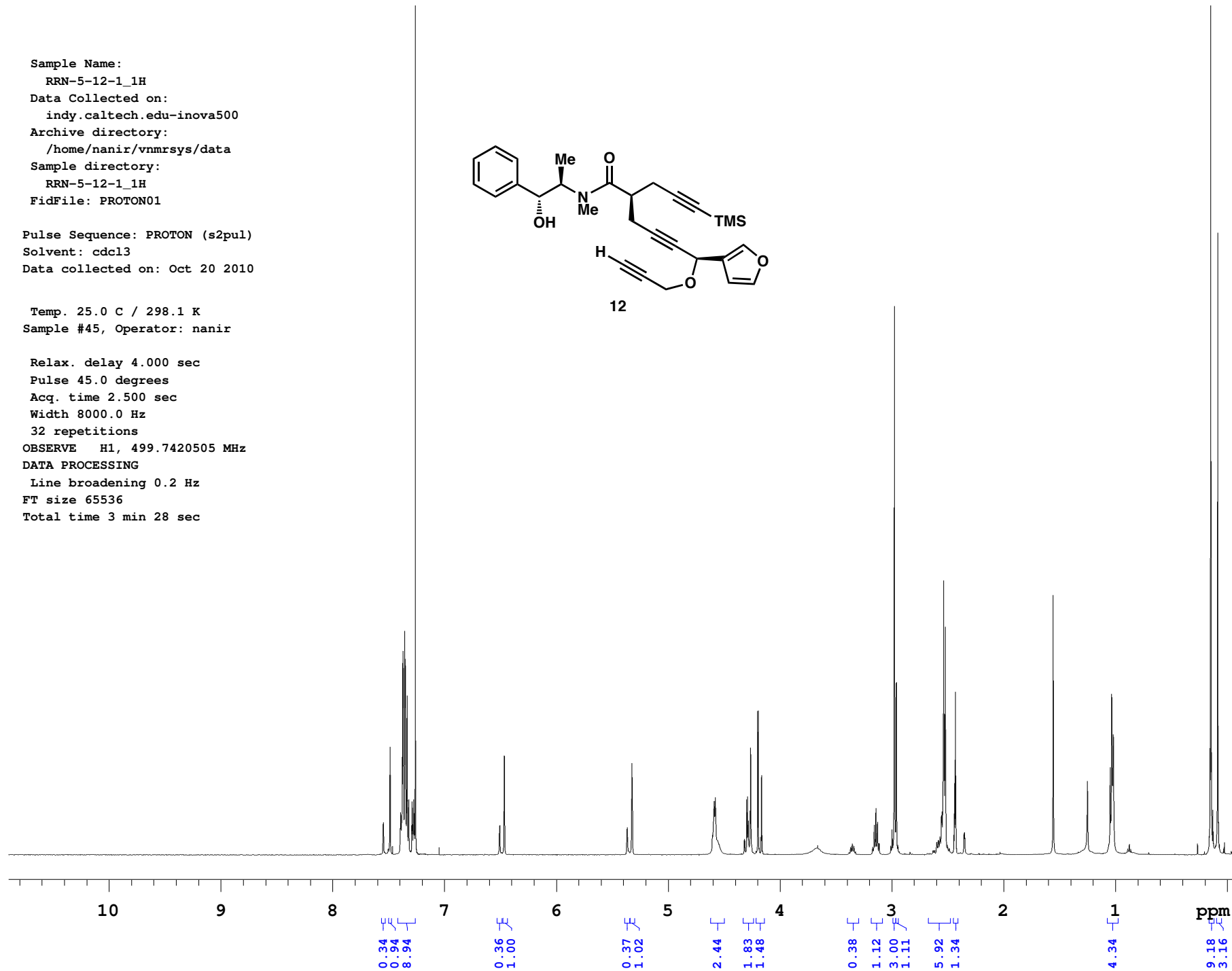
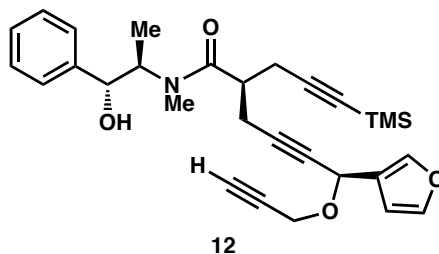


Sample Name:
 RRN-5-12-1_1H
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-5-12-1_1H
 FidFile: PROTON01

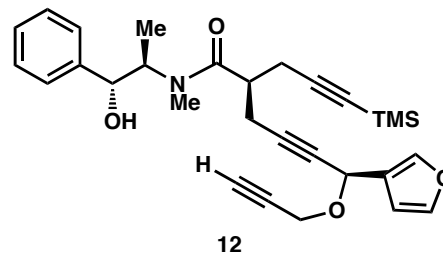
Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
 Sample #45, Operator: nanir

Relax. delay 4.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 32 repetitions
 OBSERVE H1, 499.7420505 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 3 min 28 sec



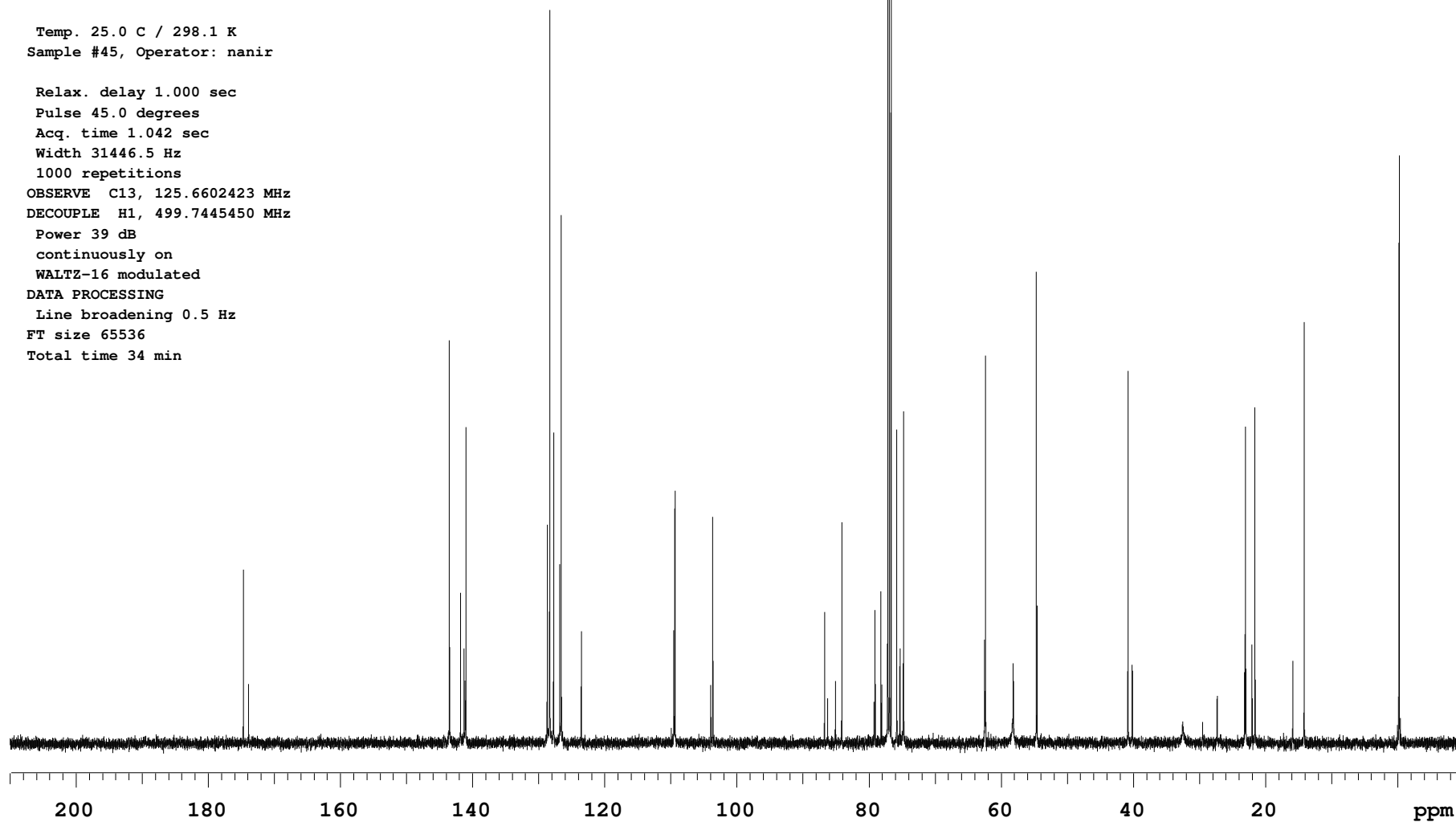
Sample Name:
RRN-5-12-1_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-5-12-1_13C
FidFile: CARBON01



Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602423 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

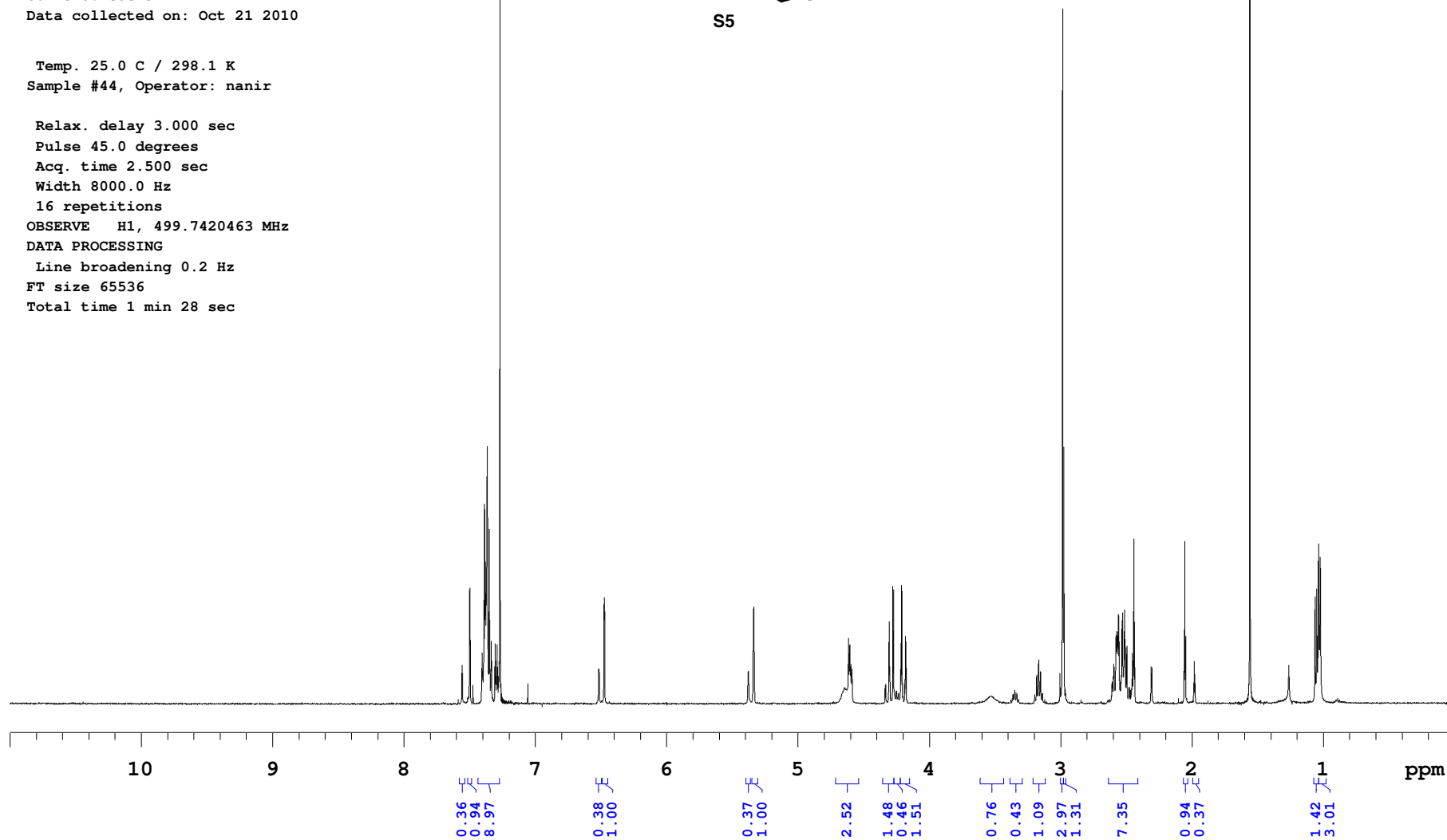
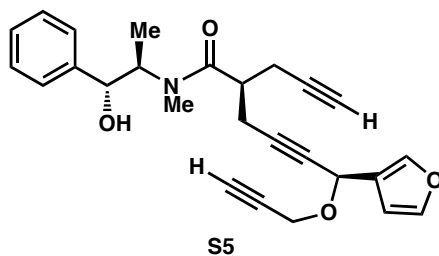


Sample Name:
 RRN-5-13-1_1H
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-5-13-1_1H
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 21 2010

Temp. 25.0 C / 298.1 K
 Sample #44, Operator: nanir

Relax. delay 3.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
 OBSERVE H1, 499.7420463 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min 28 sec

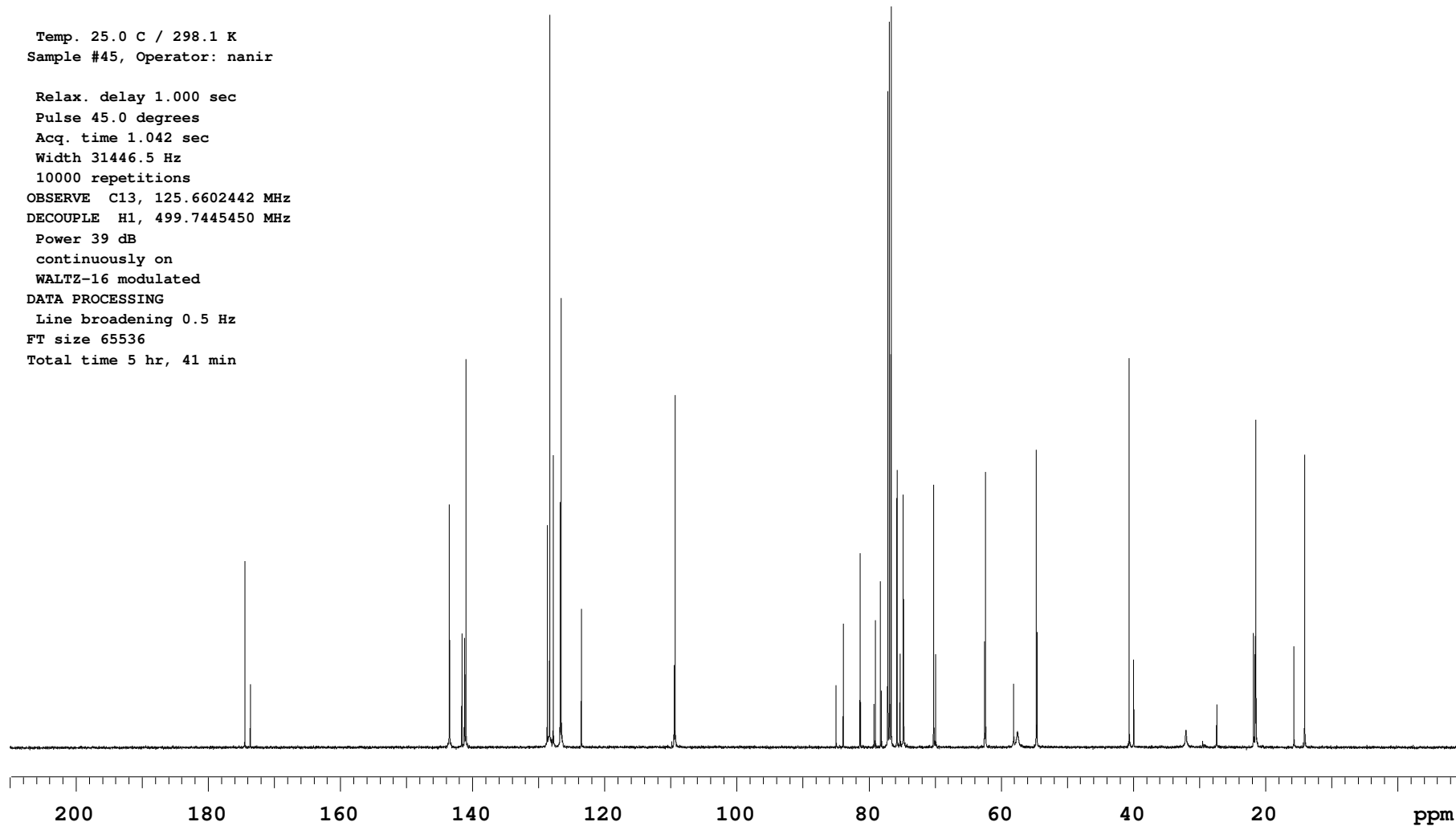
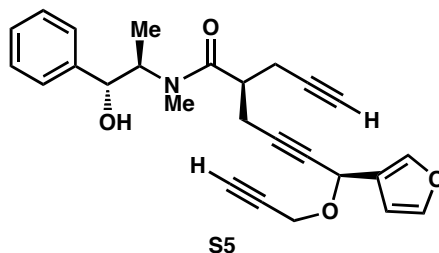


Sample Name:
RRN-5-13-1_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-5-13-1_13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 21 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
10000 repetitions
OBSERVE C13, 125.6602442 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 5 hr, 41 min

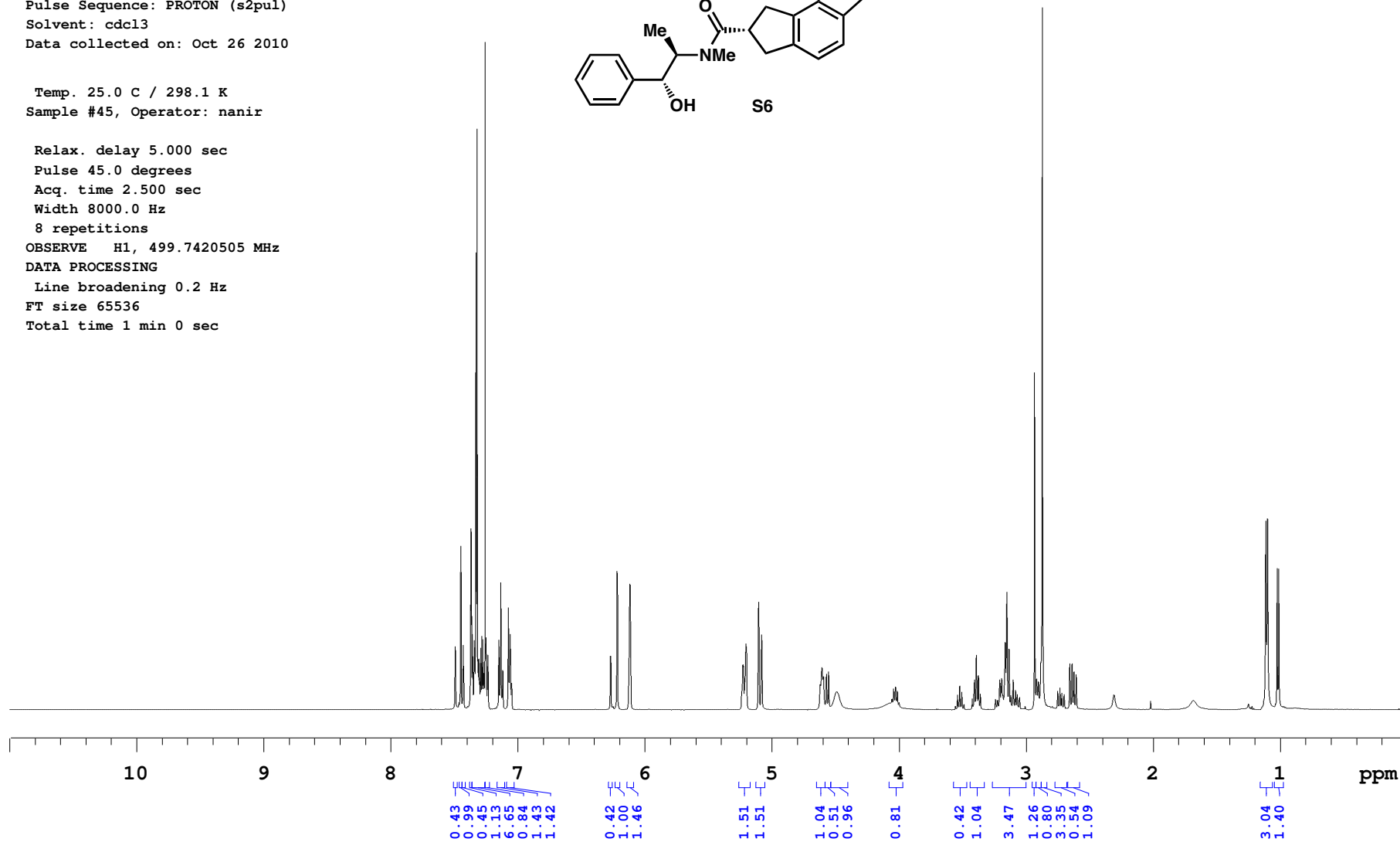
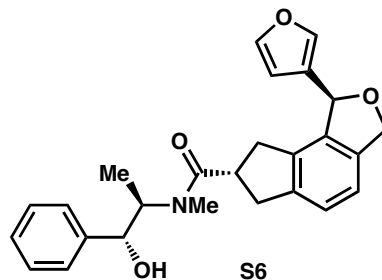


Sample Name:
 RRN-5-15-2_13C
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-5-15-2_13C
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 26 2010

Temp. 25.0 C / 298.1 K
 Sample #45, Operator: nanir

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.7420505 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min 0 sec

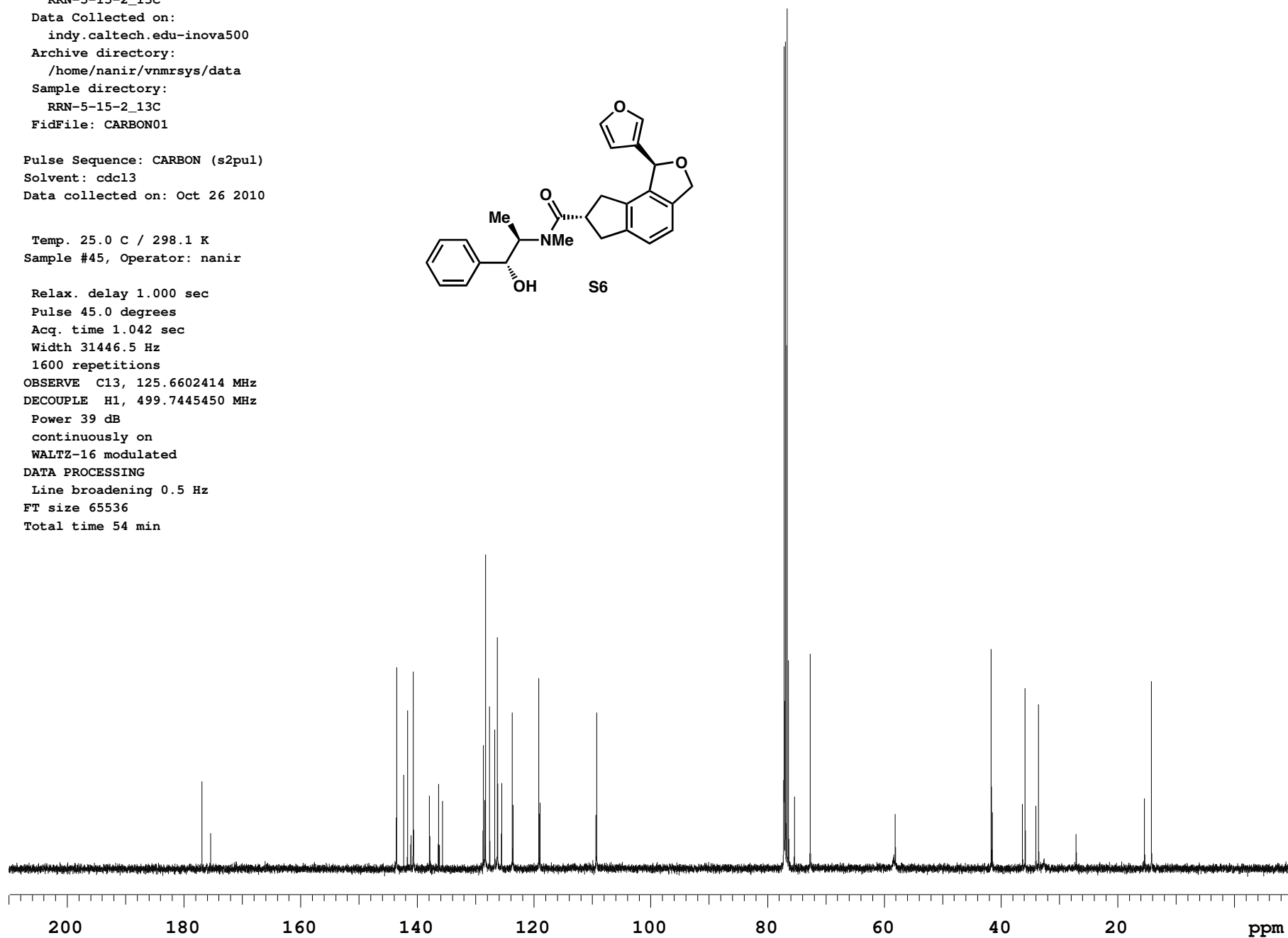
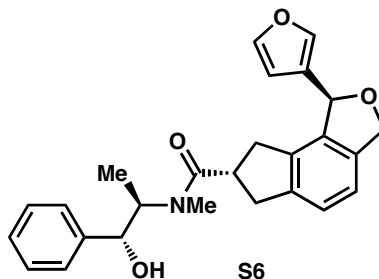


Sample Name:
RRN-5-15-2_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-5-15-2_13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 26 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1600 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 54 min

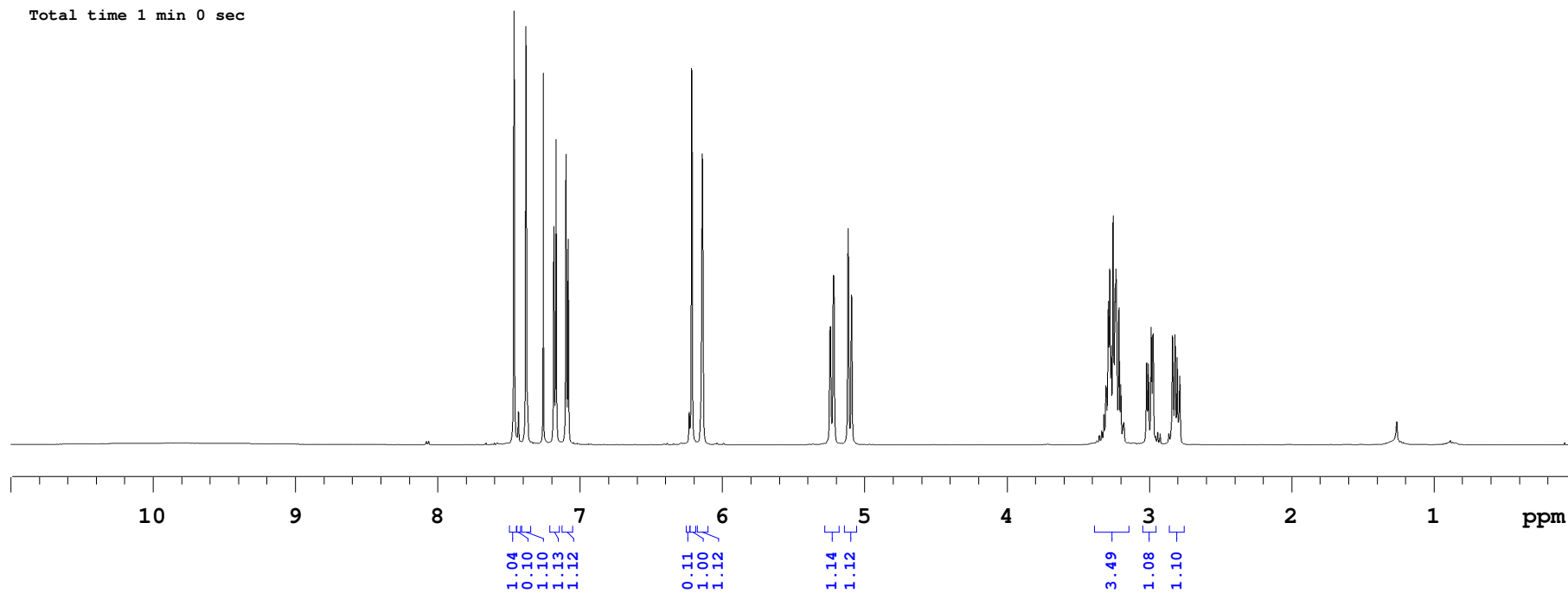
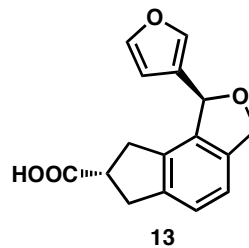


Sample Name:
RRN-4-300-1_1H_II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-300-1_1H_II
FidFile: PROTON01

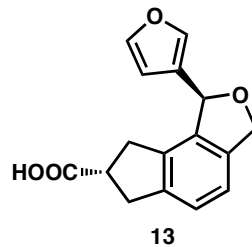
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2010

Temp. 25.0 C / 298.1 K
Sample #44, Operator: nanir

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420502 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec



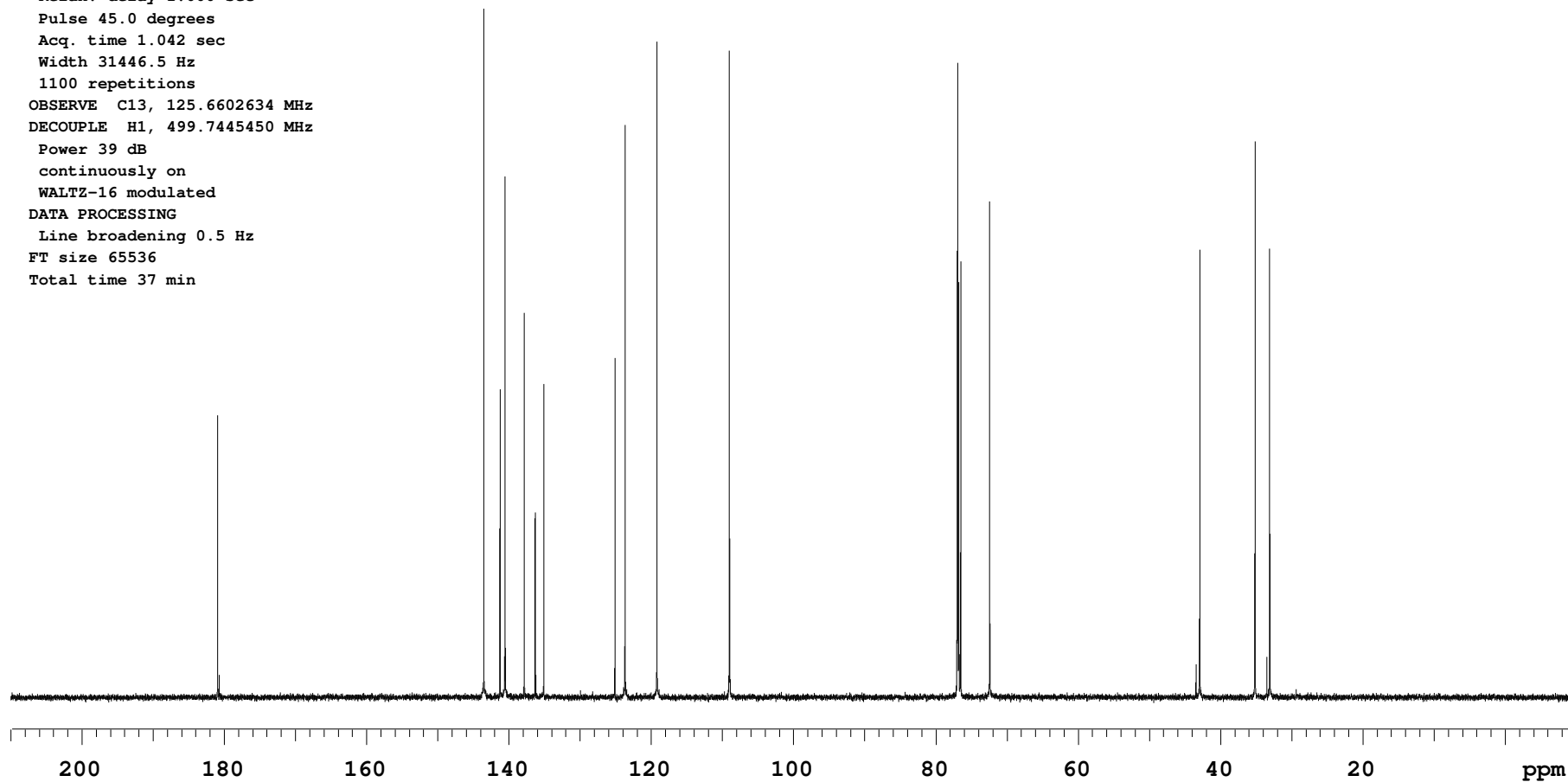
Sample Name:
RRN-4-300-13C_II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-300-13C_II
FidFile: CARBON01



Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2010

Temp. 25.0 C / 298.1 K
Sample #43, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1100 repetitions
OBSERVE C13, 125.6602634 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 37 min

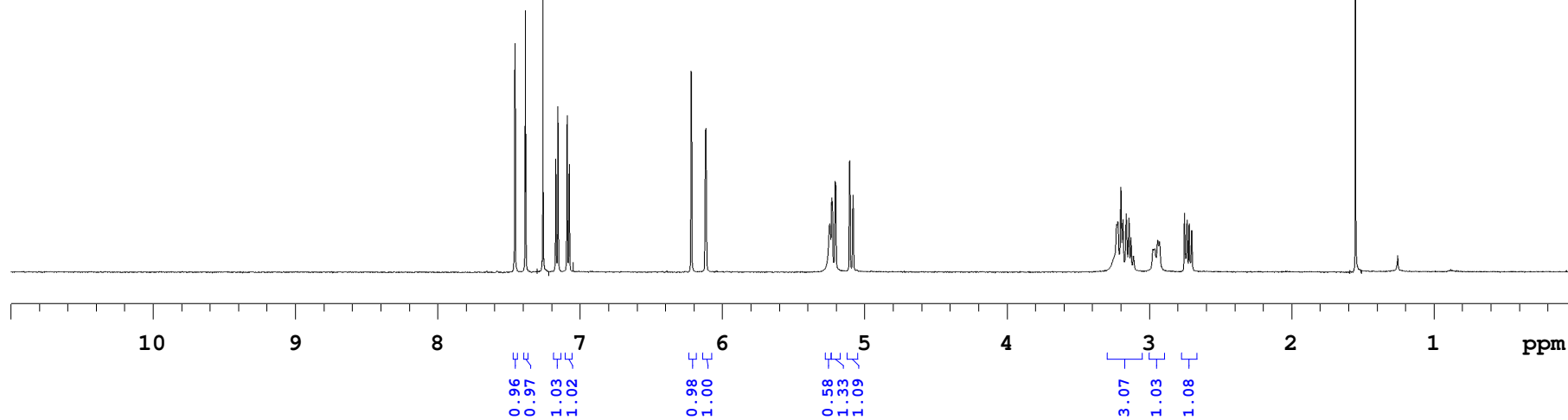
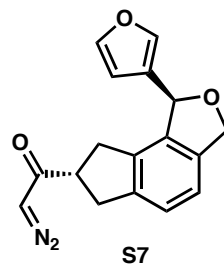


Sample Name:
RRN-4-298-1_1H_pHPLC
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-298-1_1H_pHPLC
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2010

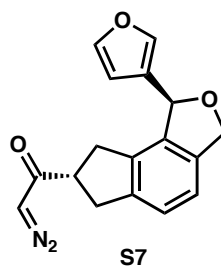
Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec



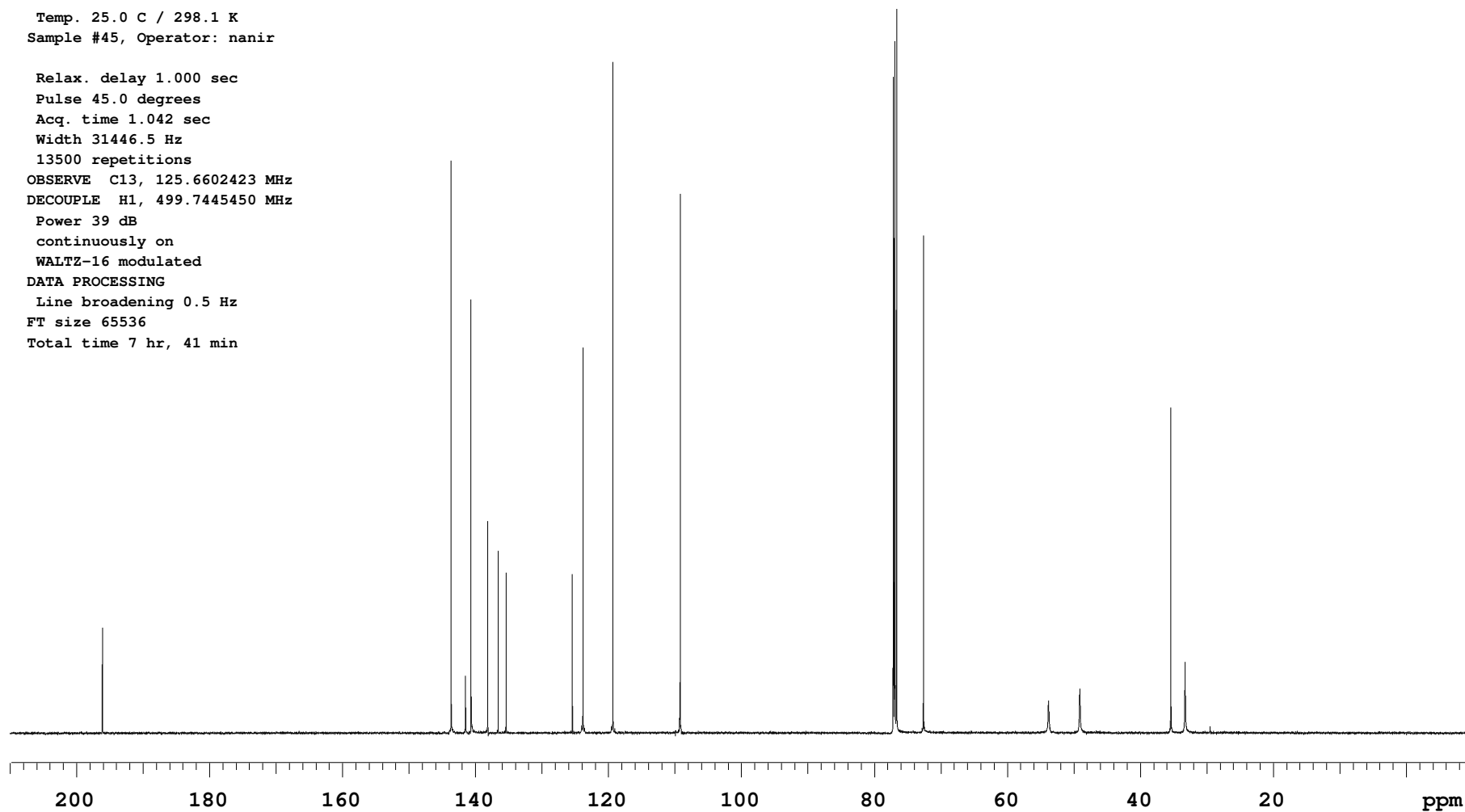
Sample Name:
RRN-4-298-pHPLC_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-298-pHPLC_13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 17 2010



Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13500 repetitions
OBSERVE C13, 125.6602423 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 41 min

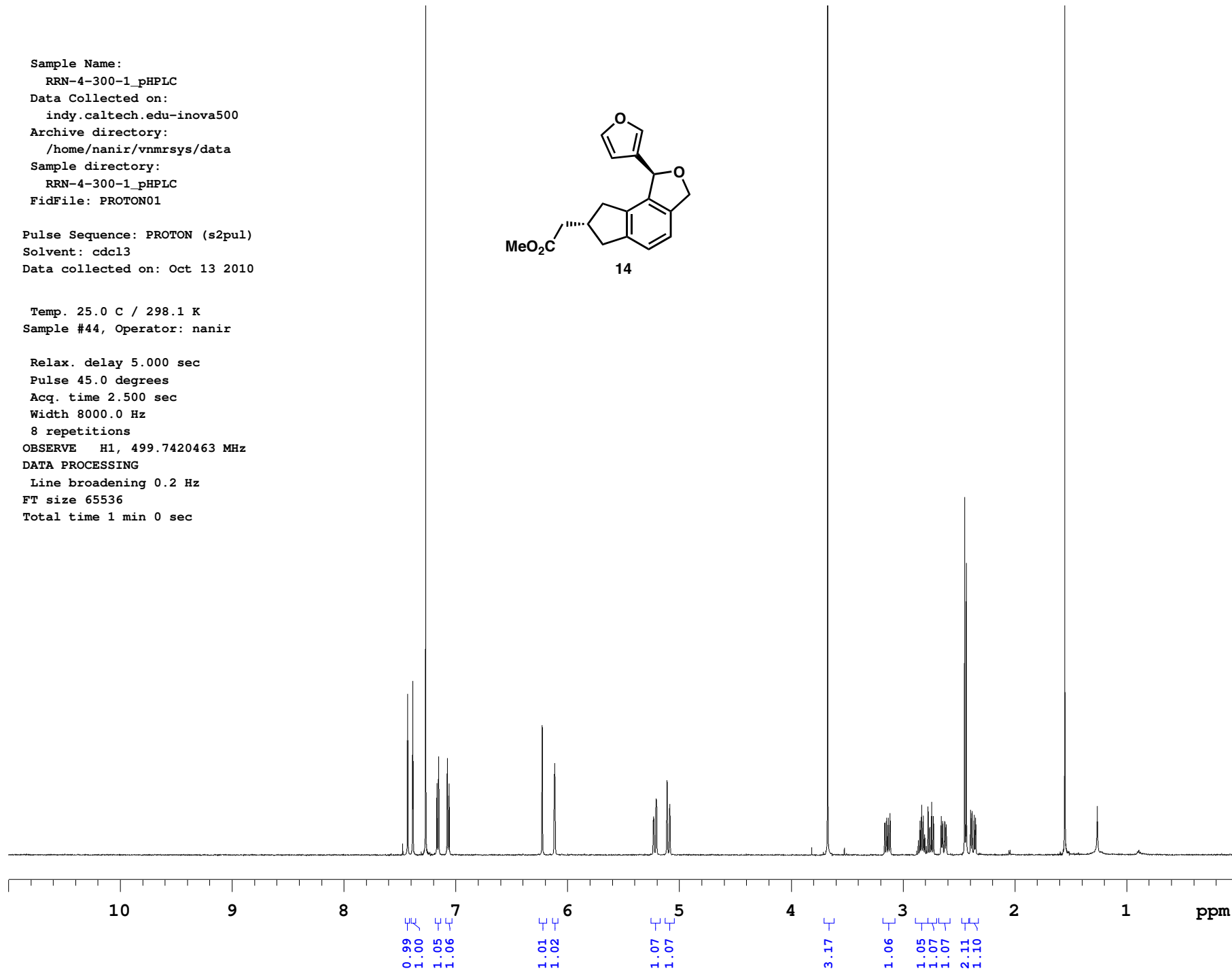
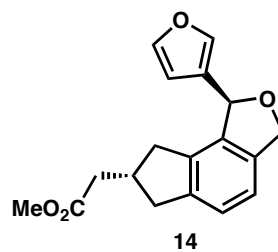


Sample Name:
 RRN-4-300-1_pHPLC
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-4-300-1_pHPLC
 FidFile: PROTON01

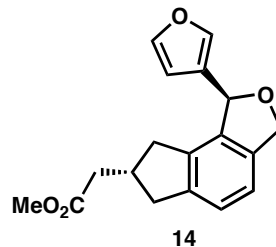
Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 13 2010

Temp. 25.0 C / 298.1 K
 Sample #44, Operator: nanir

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.7420463 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min 0 sec



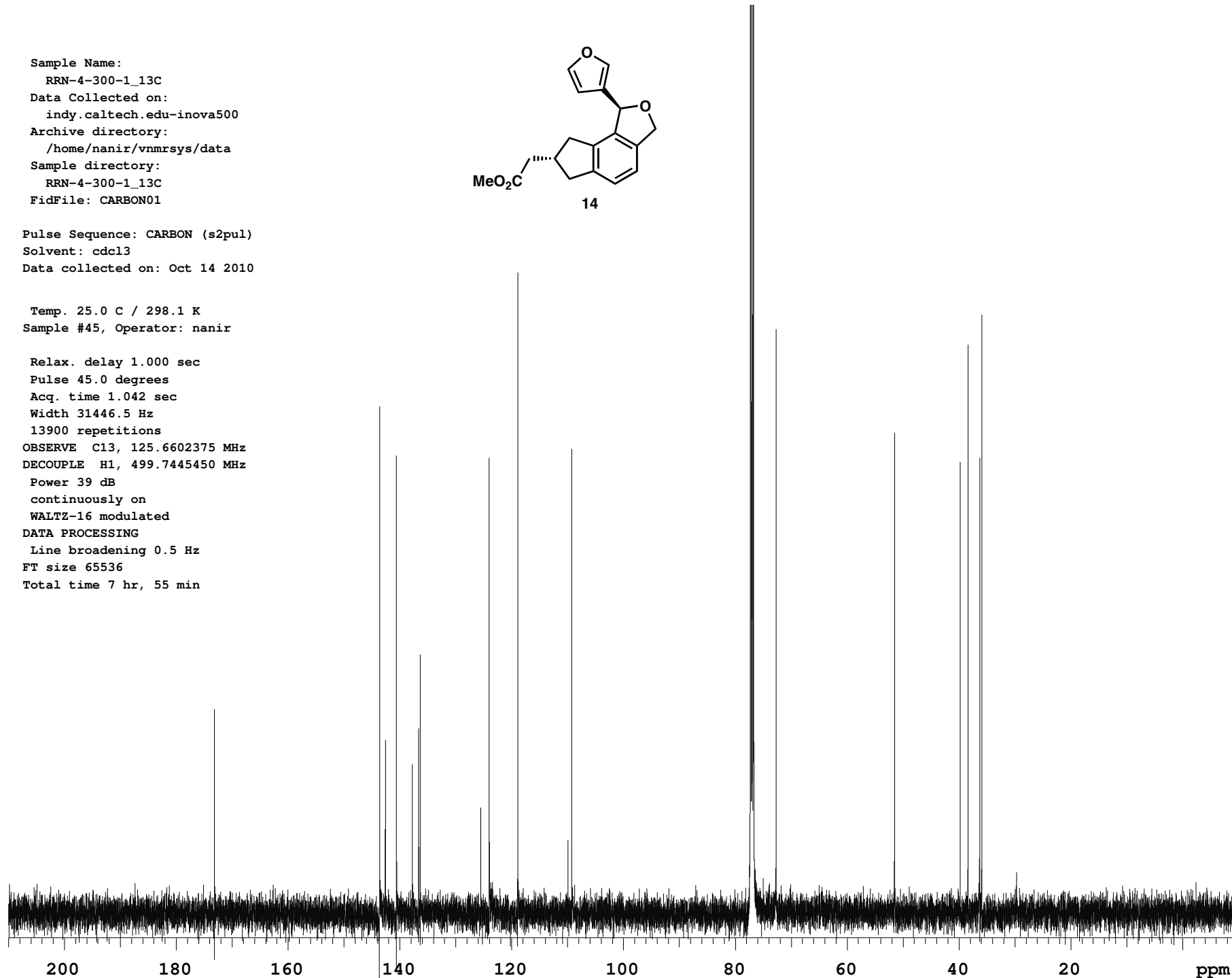
Sample Name:
RRN-4-300-1_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-4-300-1_13C
FidFile: CARBON01



Pulse Sequence: CARBON (s2pul)
Solvent: cdcl₃
Data collected on: Oct 14 2010

Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13900 repetitions
OBSERVE C13, 125.6602375 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 55 min

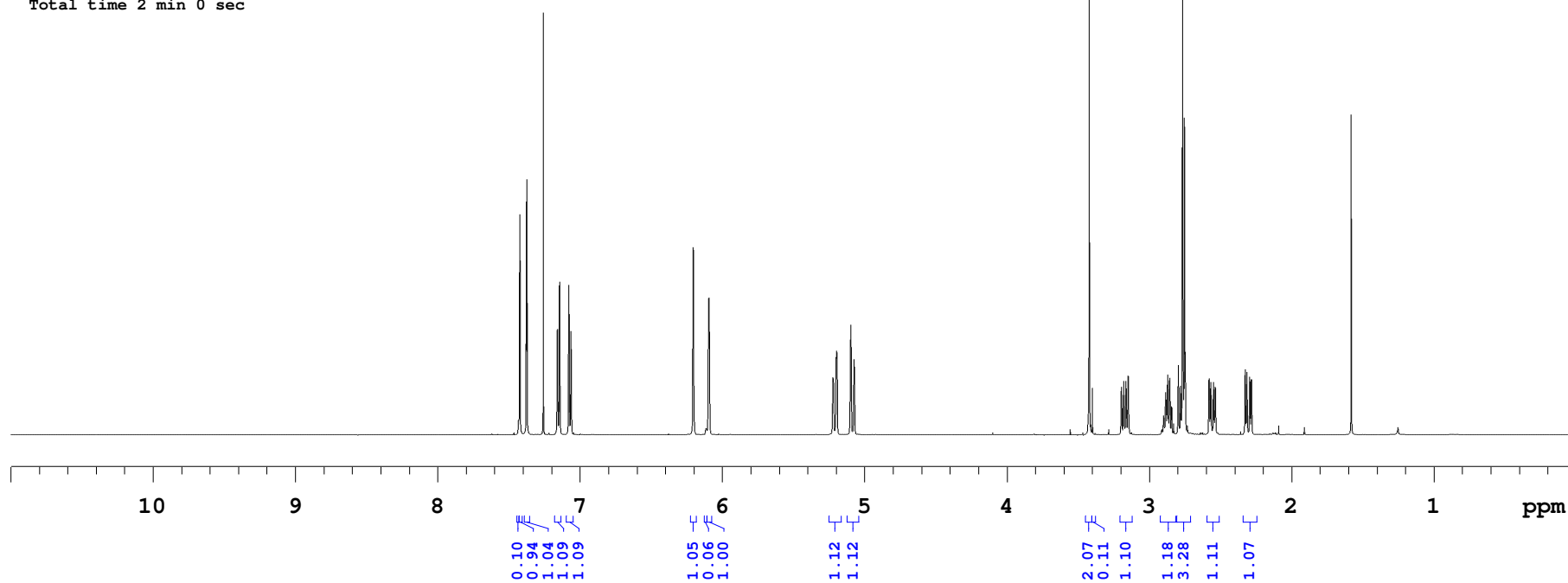
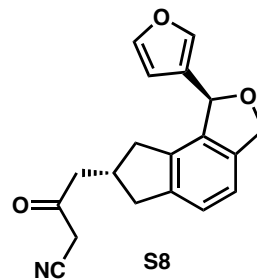


Sample Name:
 RRN-5-3-2_1H
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-5-3-2_1H
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 19 2010

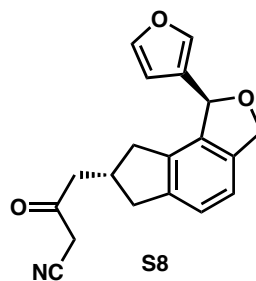
Temp. 25.0 C / 298.1 K
 Sample #44, Operator: nanir

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
 OBSERVE H1, 499.7420505 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 2 min 0 sec



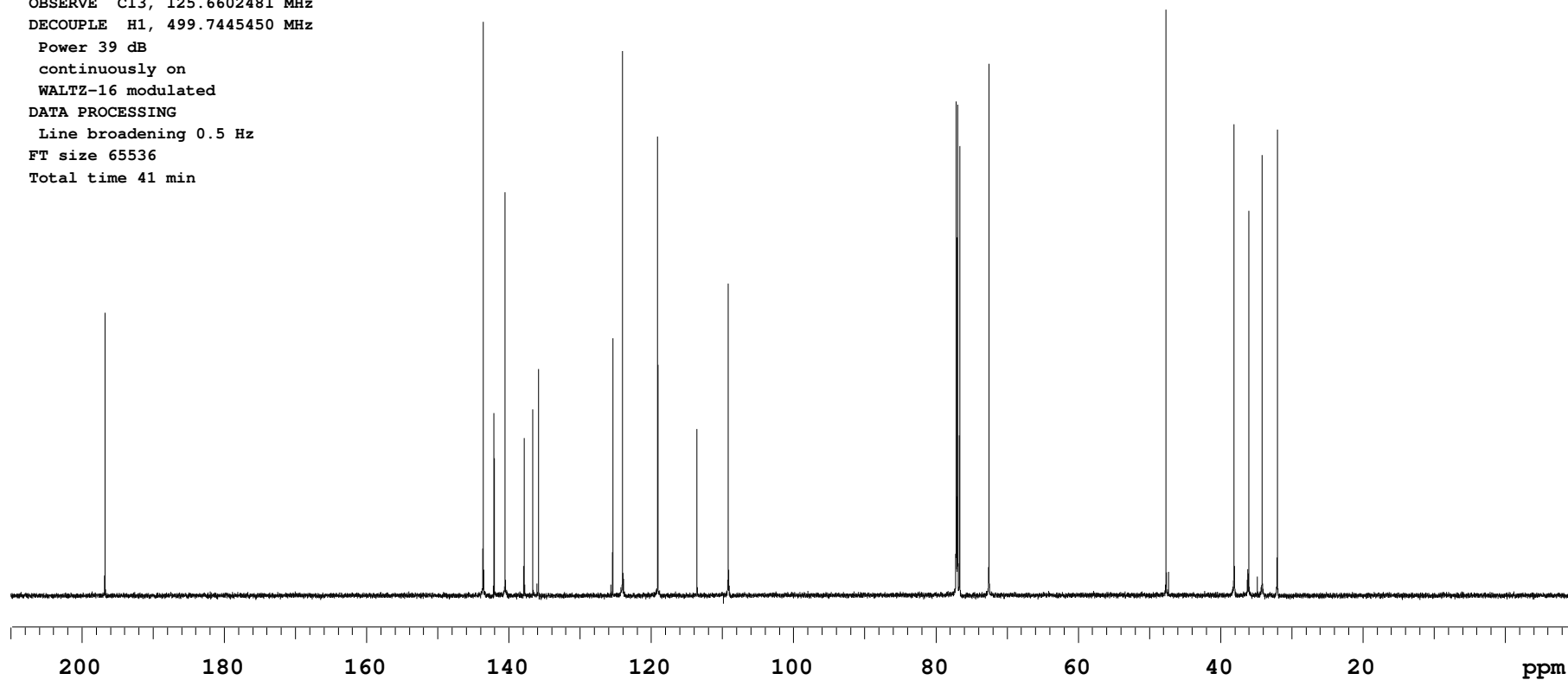
Sample Name:
RRN-5-3-2_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-5-3-2_13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 19 2010



Temp. 25.0 C / 298.1 K
Sample #45, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1200 repetitions
OBSERVE C13, 125.6602481 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 41 min

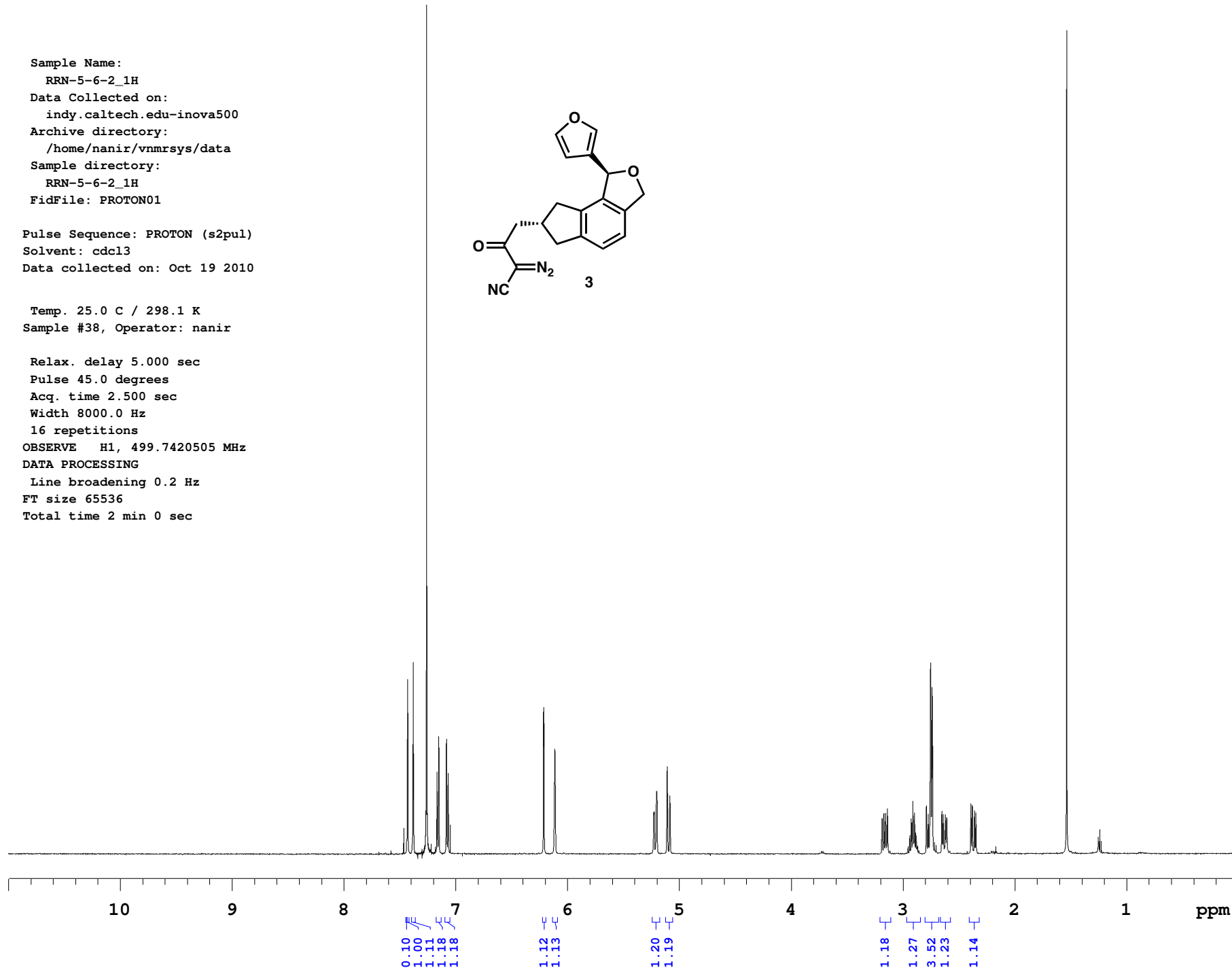
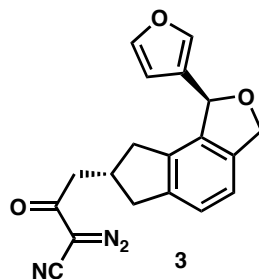


Sample Name:
 RRN-5-6-2_1H
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/nanir/vnmrsys/data
 Sample directory:
 RRN-5-6-2_1H
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 19 2010

Temp. 25.0 C / 298.1 K
 Sample #38, Operator: nanir

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
 OBSERVE H1, 499.7420505 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 2 min 0 sec

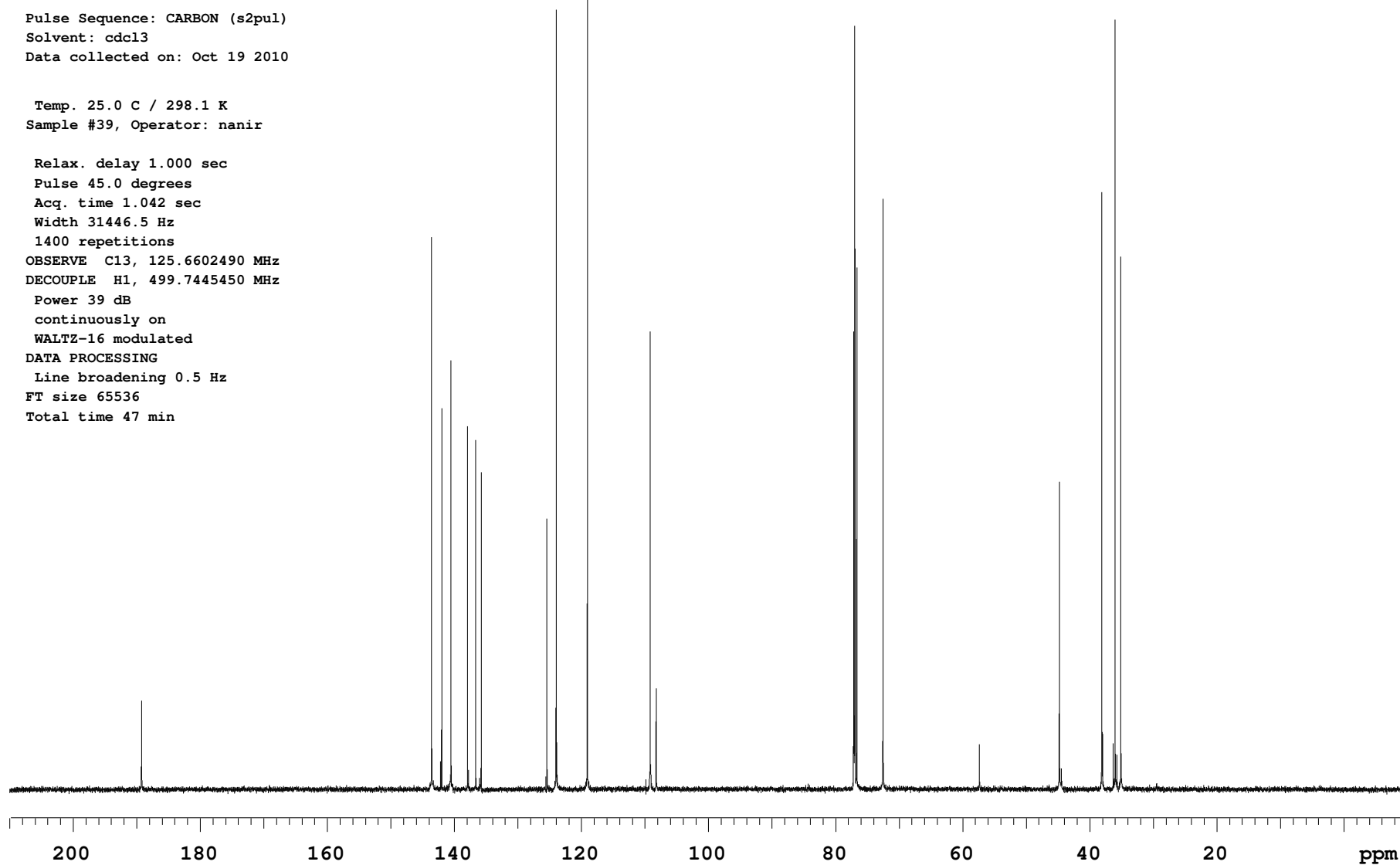
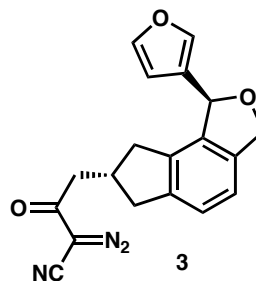


Sample Name:
RRN-5-6-2_13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nanir/vnmrsys/data
Sample directory:
RRN-5-6-2_13C
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 19 2010

Temp. 25.0 C / 298.1 K
Sample #39, Operator: nanir

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602490 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min



SL-5-252-column-600-H

Sample Name:

SL-5-252-pHPLC-F1-600-H

Data Collected on:

fid.caltech.edu-inova600

Archive directory:

Sample directory:

FidFile: SL-5-252-pHPLC-F1-600-H

Pulse Sequence: Proton (s2pul)

Solvent: cdcl3

Data collected on: Aug 20 2010

Temp. 25.0 C / 298.1 K

Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 9594.6 Hz

8 repetitions

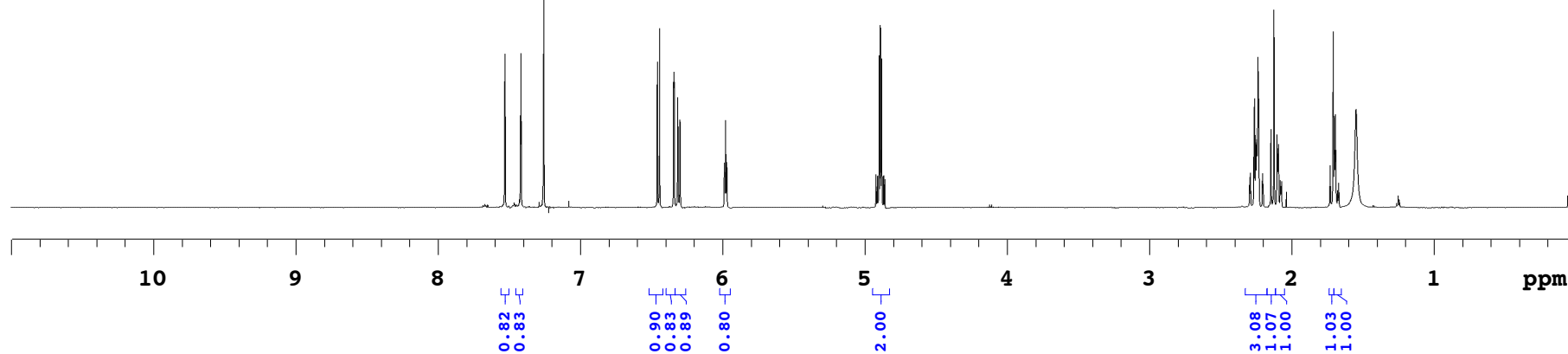
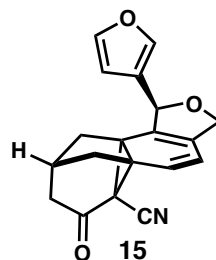
OBSERVE H1, 599.6604887 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 0 min 38 sec



Sample Name:

SL-5-249-F1-pHPLC-500-13C

Data Collected on:

indy.caltech.edu-inova500

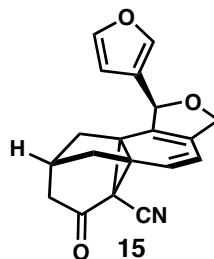
Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-5-249-F1-pHPLC-500-13C

FidFile: CARBON01



Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Aug 23 2010

Temp. 25.0 C / 298.1 K

Sample #48, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

13500 repetitions

OBSERVE C13, 125.6602389 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

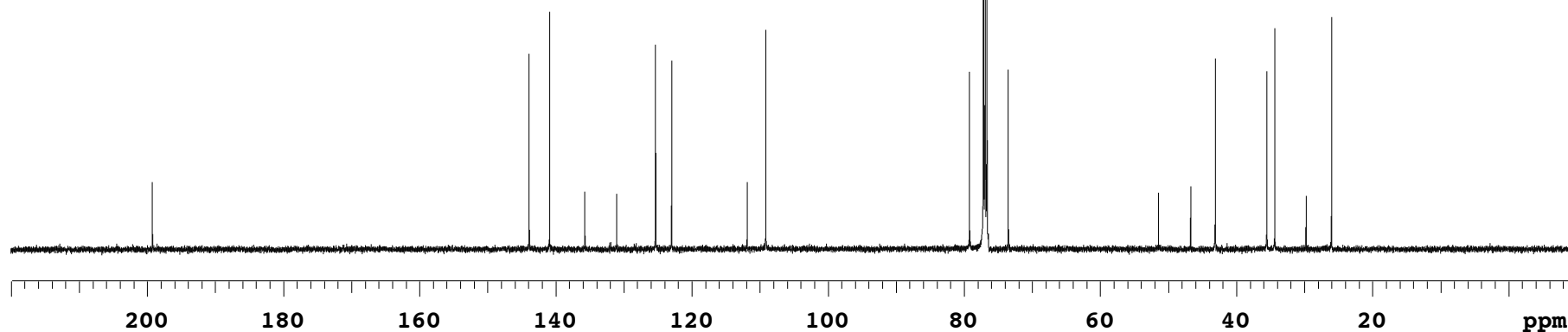
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 7 hr, 41 min



SL-6-002-column-500-H

Sample Name:

SL-6-002-column-500-H

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-002-column-500-H

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Sep 5 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

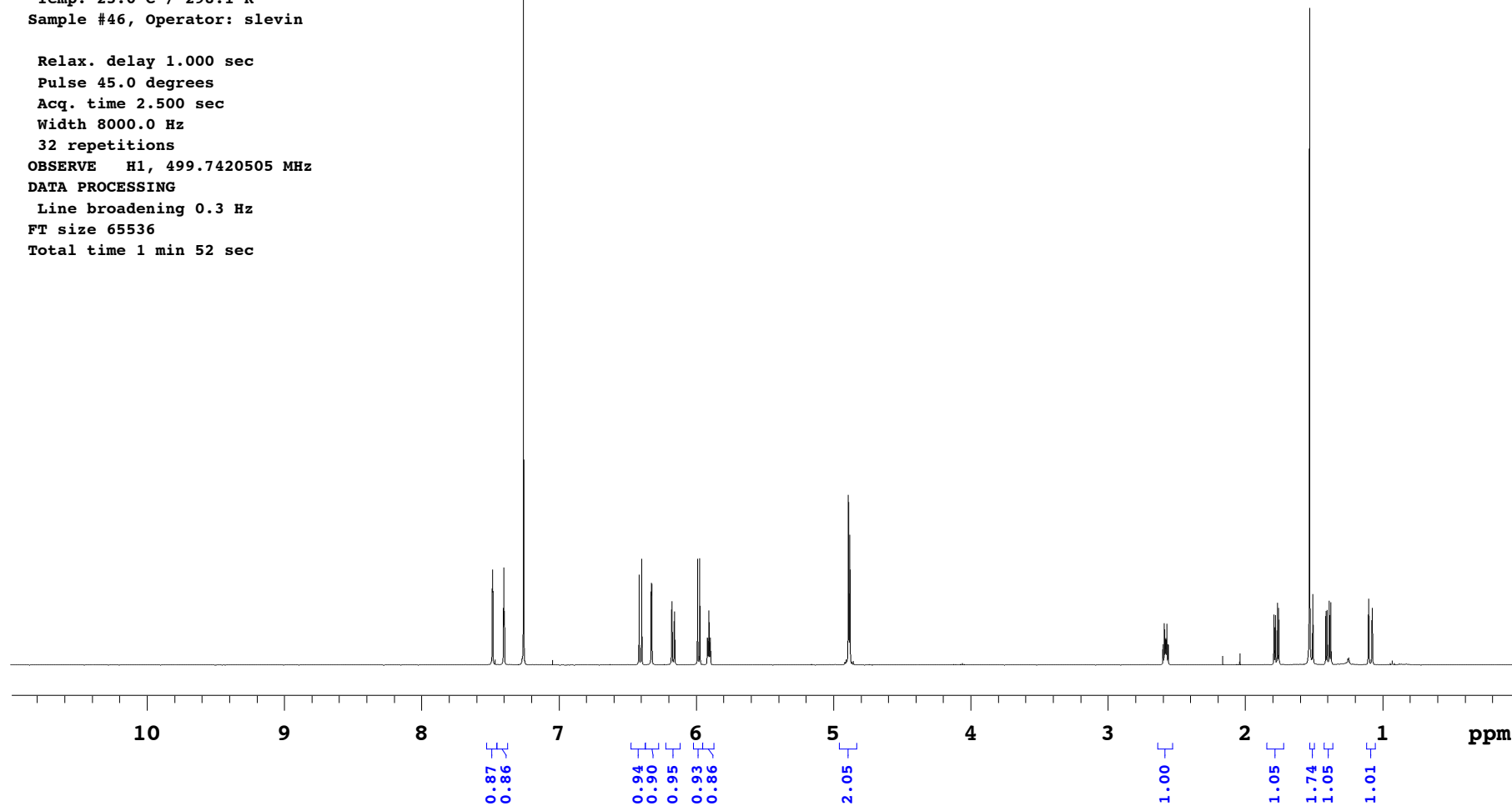
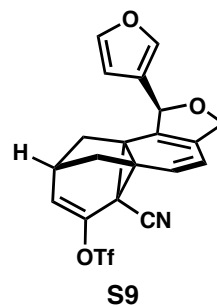
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 52 sec



SL-6-002-column-125-13C

Sample Name:

SL-6-002-column-125-13C

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-002-column-125-13C

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Sep 5 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1500 repetitions

OBSERVE C13, 125.6602390 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

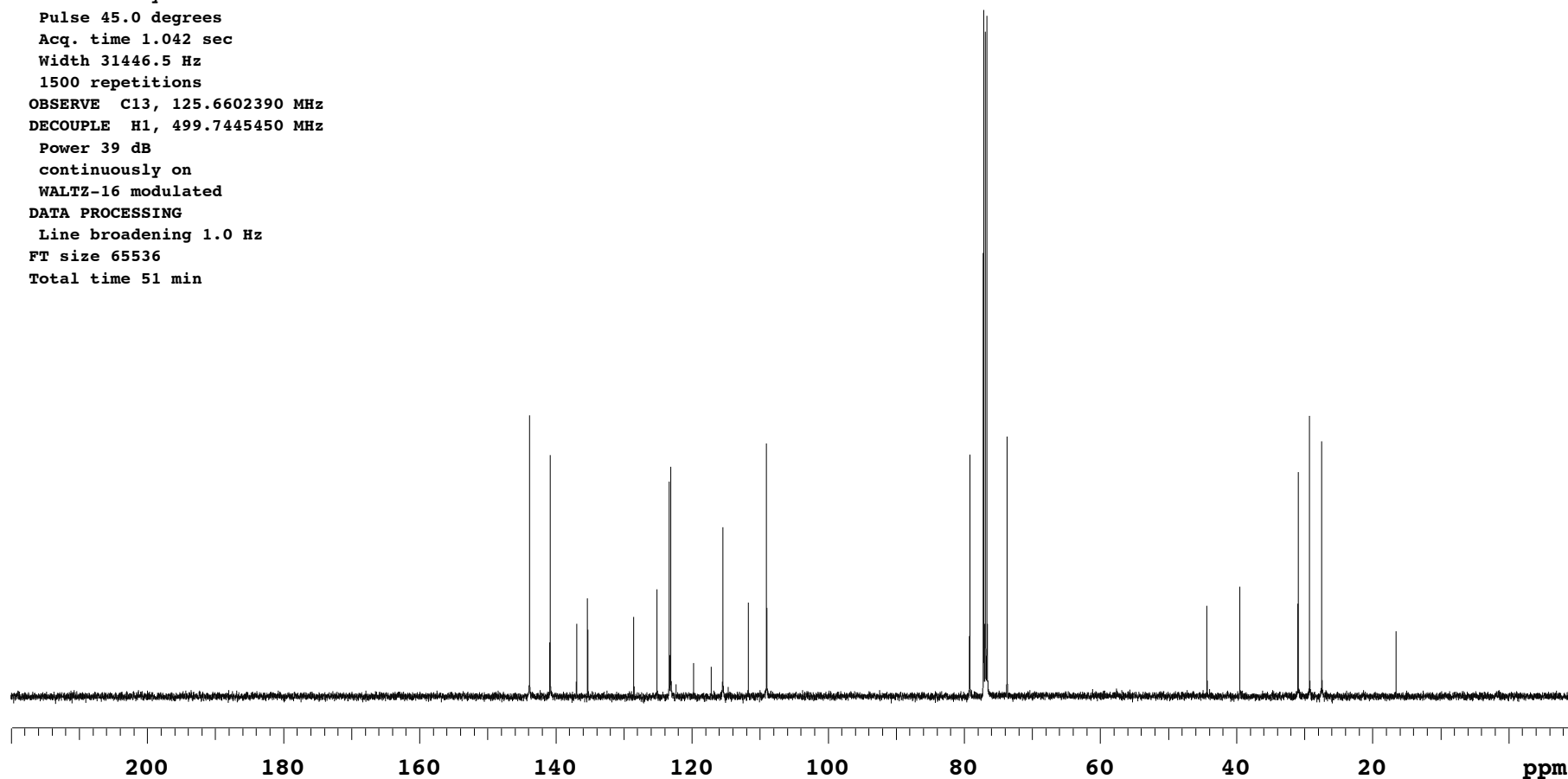
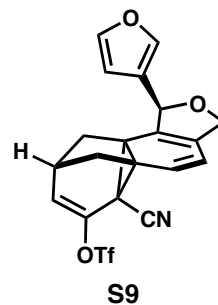
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 51 min



S59

SL-6-004-pHPLC-600-512-H

Sample: SL-6-004-pHPLC-600-512-H

File: home/slevin/SL-6-004-pHPLC-600-H.fid

Pulse Sequence: s2pul

Solvent: cd2cl2

Temp. 25.0 C / 298.1 K

Operator: slevin

File: SL-6-004-pHPLC-600-H

INOVA-600 "fid.caltech.edu"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 9594.6 Hz

512 repetitions

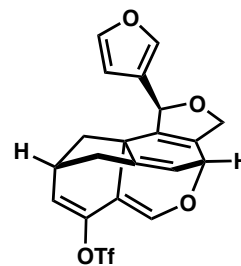
OBSERVE H1, 599.6616487 MHz

DATA PROCESSING

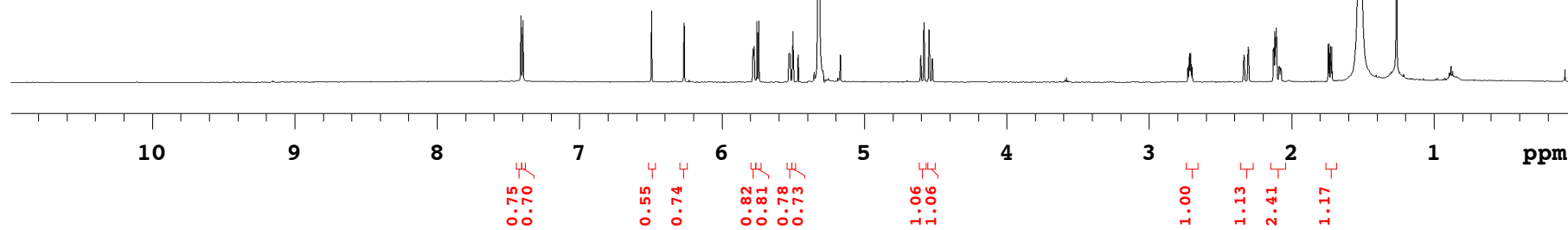
Line broadening 0.3 Hz

FT size 65536

Total time 26 min, 12 sec



17



SL-6-085-crude-500-H

Sample Name:

SL-6-085-crude-500-H

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

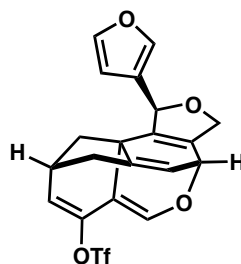
SL-6-085-crude-500-H

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Nov 9 2010



17

125 MHz ^{13}C of 17
without purification

Temp. 25.0 C / 298.1 K

Sample #47, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

320 repetitions

OBSERVE C13, 125.6604283 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

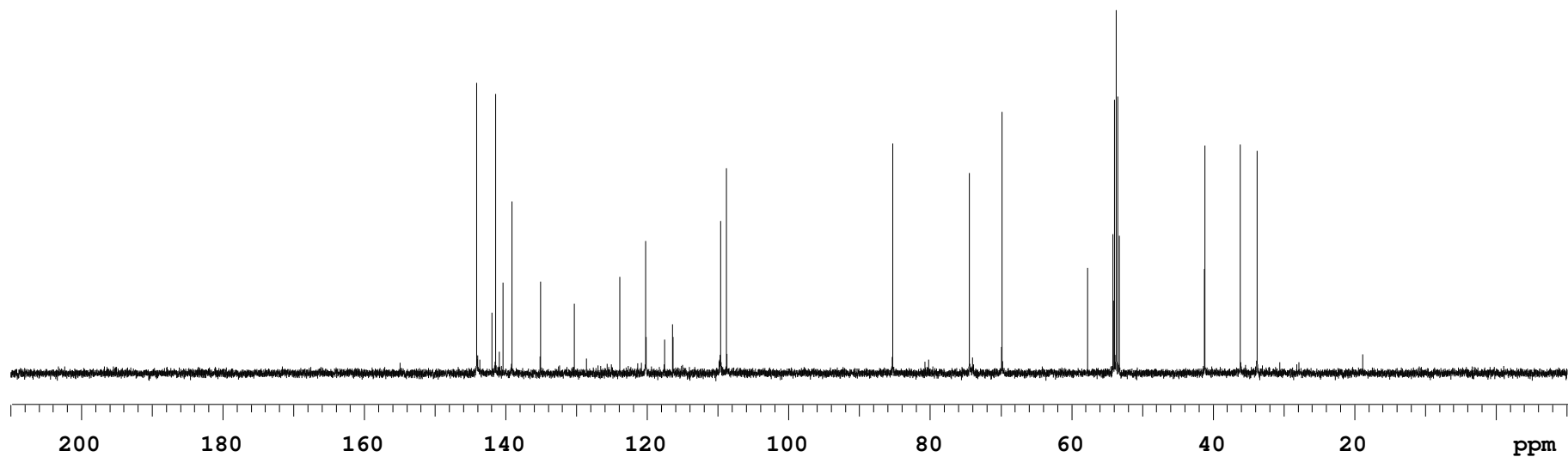
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 1 hr, 8 min



S61

SL-6-007-column-500-H

Sample Name:

SL-6-007-column-500-H

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-007-column-500-H

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Sep 7 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

128 repetitions

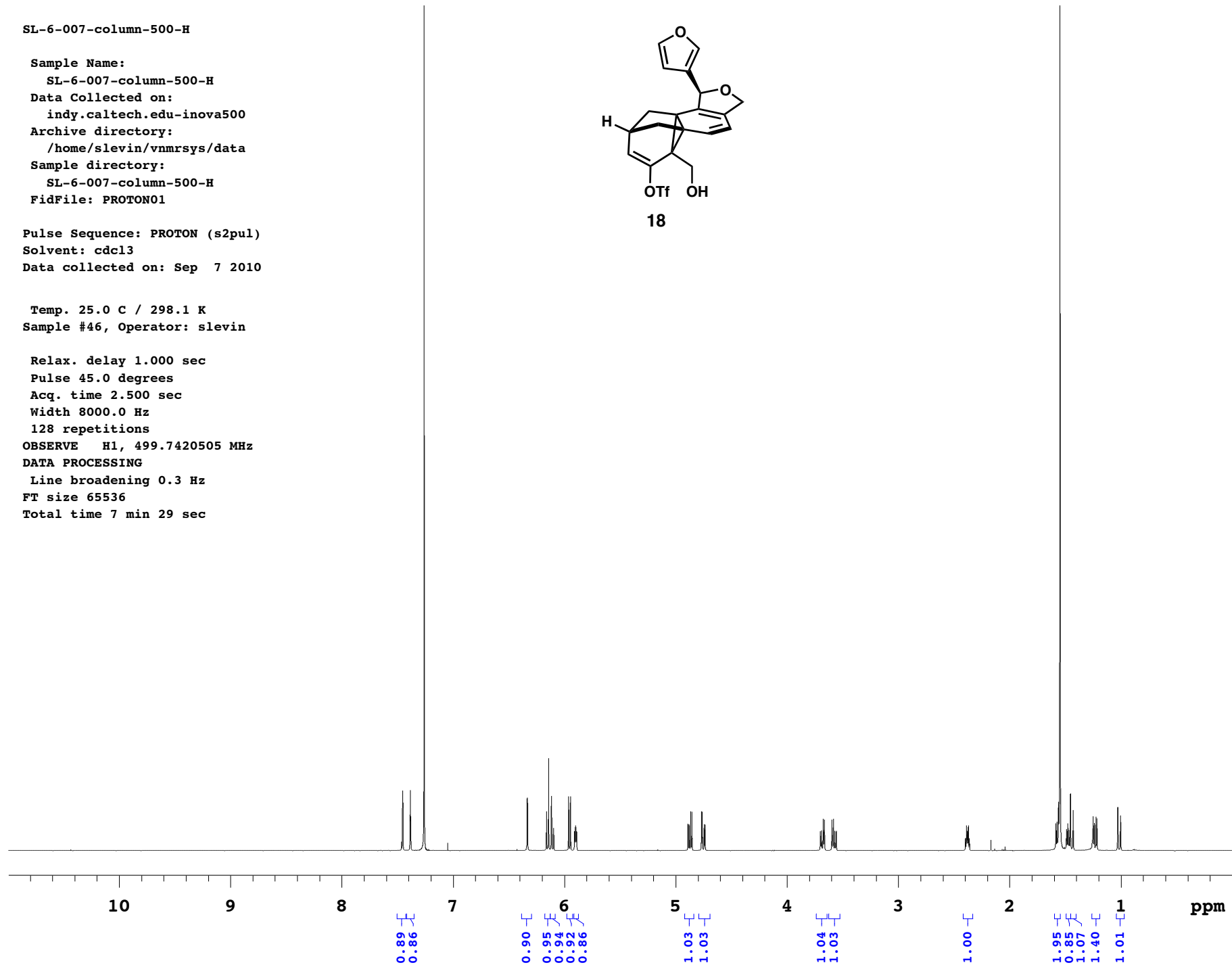
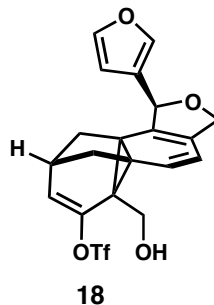
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 7 min 29 sec



SL-6-007-column-125-13C

Sample Name:

SL-6-007-column-125-13C

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-007-column-125-13C

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Sep 8 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

13500 repetitions

OBSERVE C13, 125.6602375 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

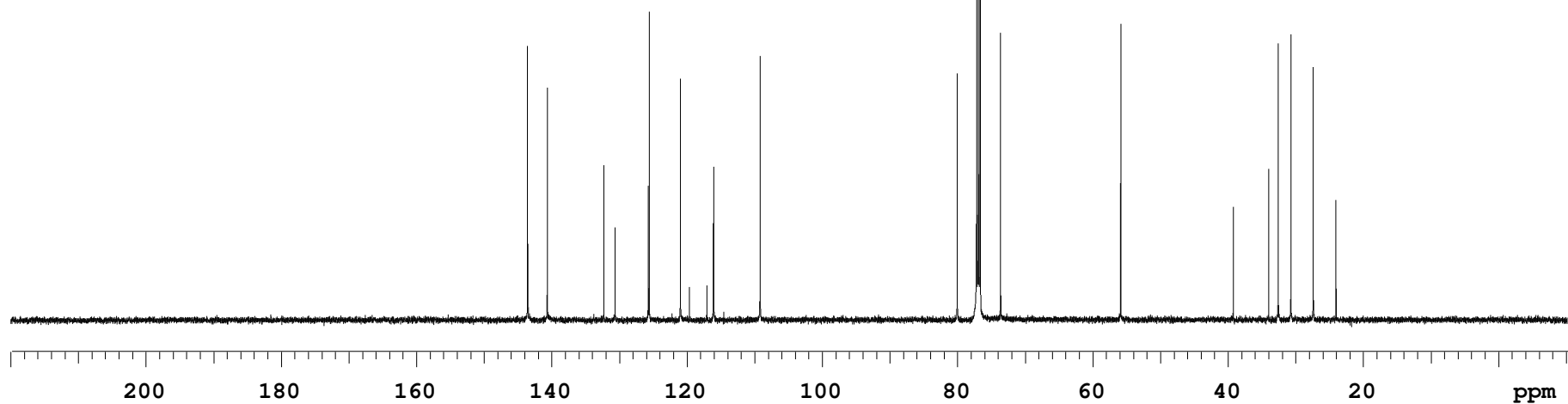
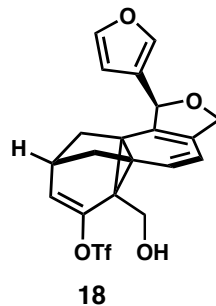
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 7 hr, 41 min



SL-5-289-column-500-H

Sample Name:

SL-5-289-column-500-H

Data Collected on:

indy.caltech.edu-inova500

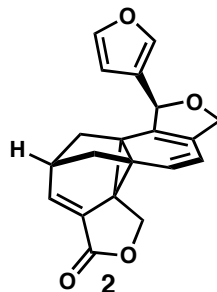
Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-5-289-column-500-H

FidFile: PROTON01



Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Aug 28 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

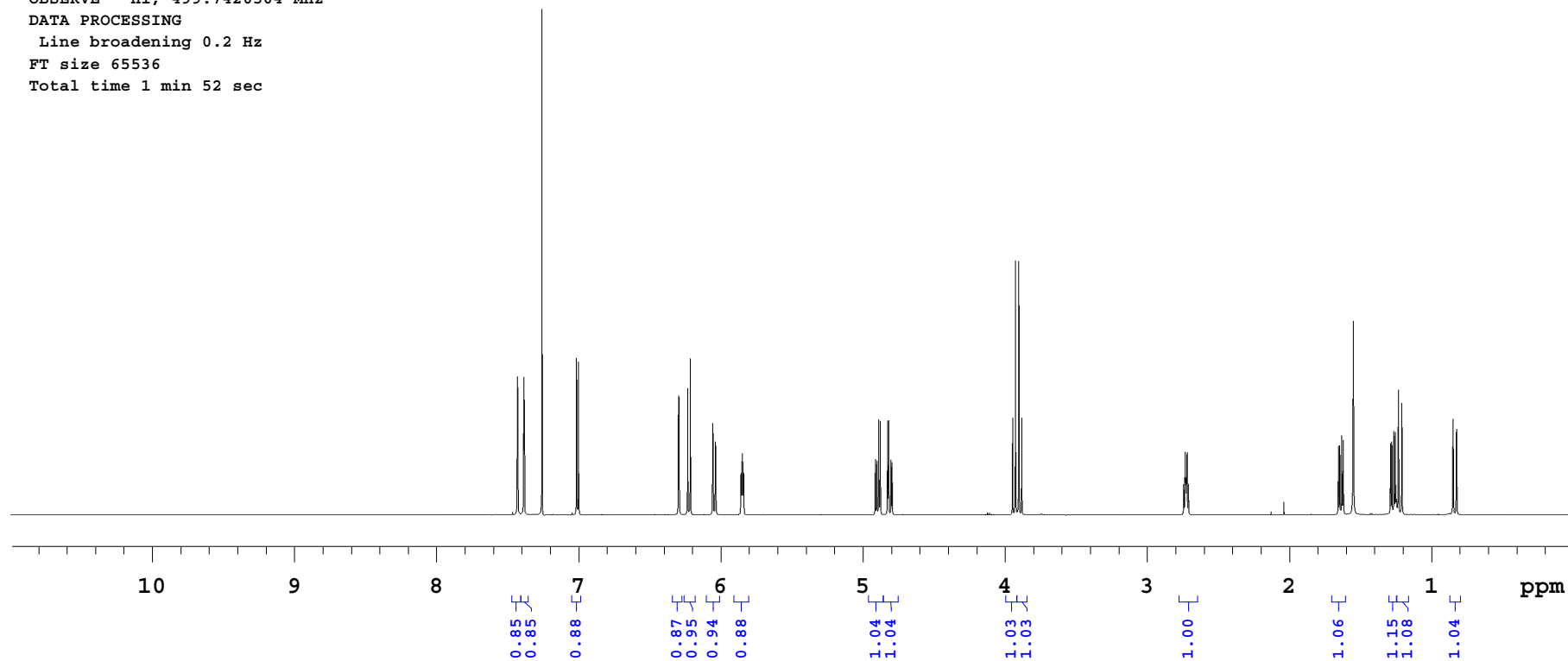
OBSERVE H1, 499.7420504 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 52 sec



SL-5-289-column-125-13C

Sample Name:

SL-5-289-column-125-13C

Data Collected on:

indy.caltech.edu-inova500

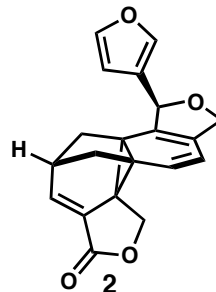
Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-5-289-column-125-13C

FidFile: CARBON01



Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Aug 28 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

2000 repetitions

OBSERVE C13, 125.6602375 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

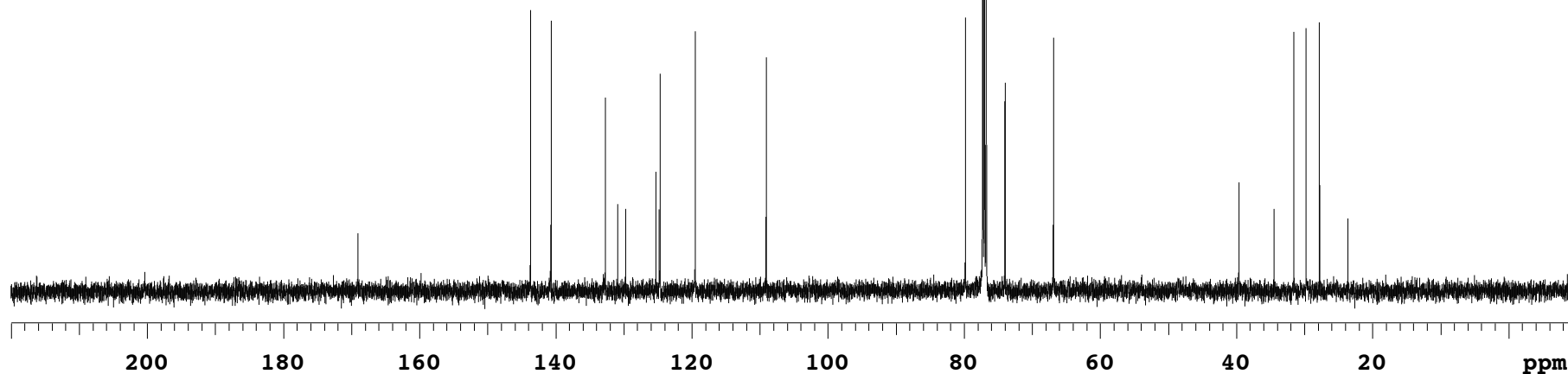
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 8 min



SL-6-054-column-500-H

Sample Name:

SL-6-054-column-500-H

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-054-column-500-H

FidFile: current

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Oct 24 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

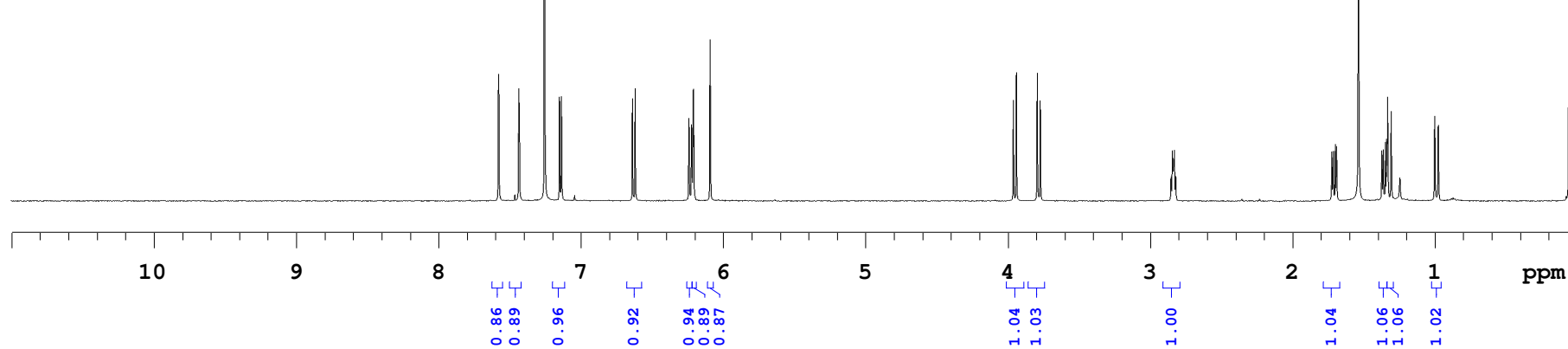
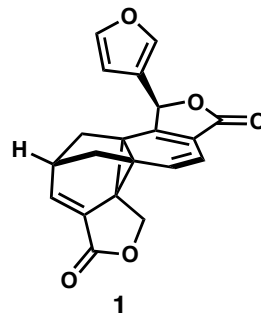
OBSERVE H1, 499.7420507 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 14 min



SL-6-067-column-125-13C

Sample Name:

SL-6-067-column-125-13C

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-067-column-125-13C

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Nov 1 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

13500 repetitions

OBSERVE C13, 125.6602385 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

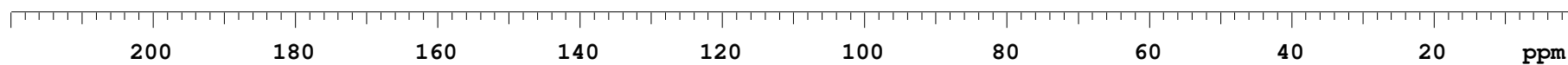
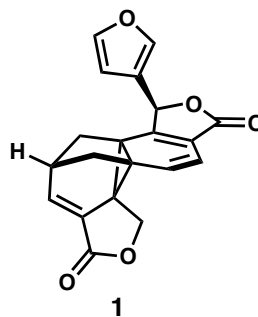
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 7 hr, 41 min



SL-6-054-ald-column-500-H

Sample Name:

SL-6-054-ald-column-500-H

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-054-ald-column-500-H

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Oct 24 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

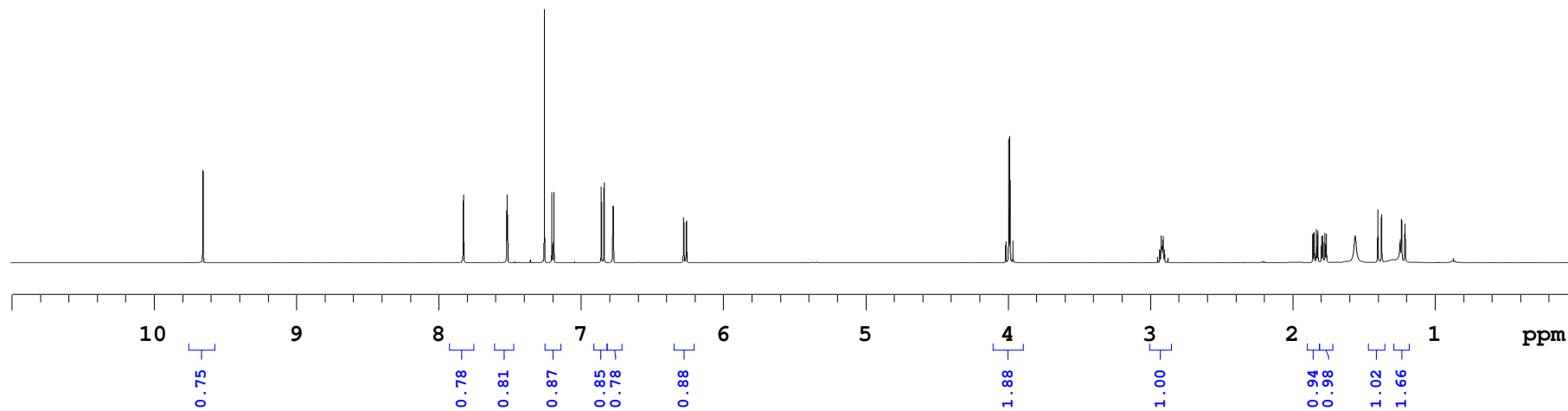
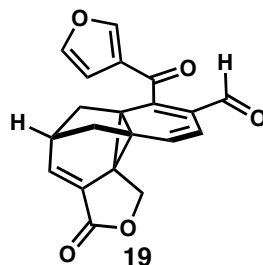
OBSERVE H1, 499.7420506 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 52 sec



SL-6-054-ald-column-500-H

Sample Name:

SL-6-054-ald-column-500-H

Data Collected on:

indy.caltech.edu-inova500

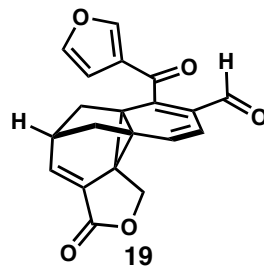
Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-054-ald-column-500-H

FidFile: CARBON01



Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Oct 24 2010

Temp. 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1550 repetitions

OBSERVE C13, 125.6602385 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

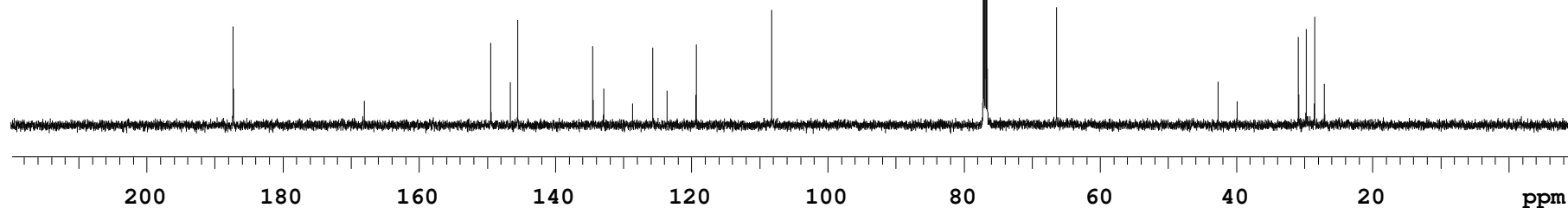
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 52 min



SL-6-004-pHPLC-600-512-H

Sample: SL-6-004-pHPLC-600-512-H

File: home/slevin/SL-6-004-pHPLC-600-H.fid

Pulse Sequence: s2pul

Solvent: cd2cl2

Temp. 25.0 C / 298.1 K

Operator: slevin

File: SL-6-004-pHPLC-600-H

INOVA-600 "fid.caltech.edu"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 9594.6 Hz

512 repetitions

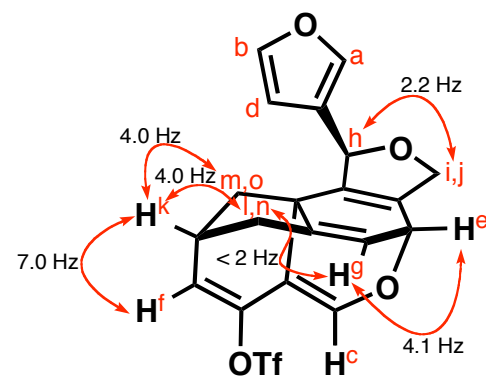
OBSERVE H1, 599.6616487 MHz

DATA PROCESSING

Line broadening 0.3 Hz

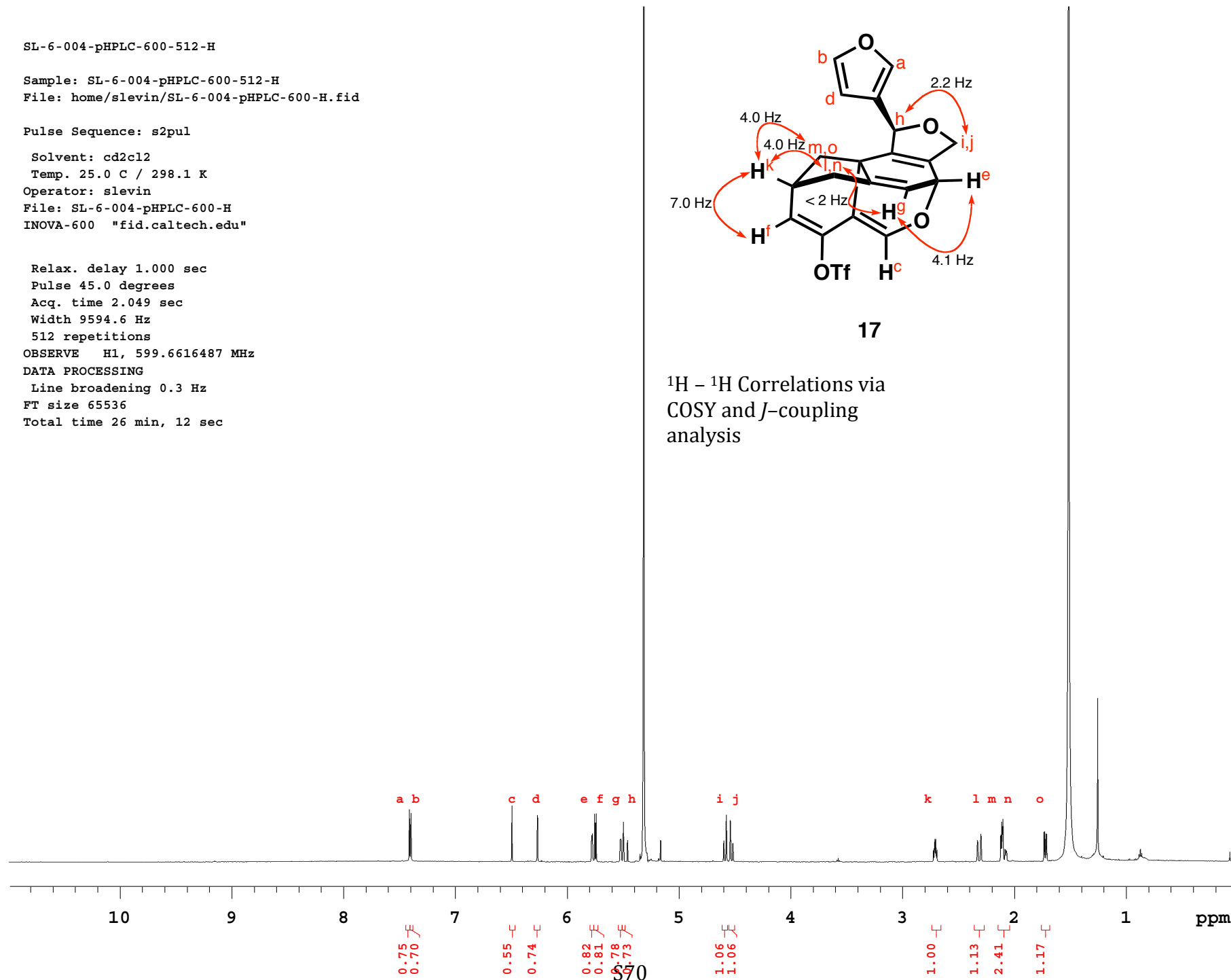
FT size 65536

Total time 26 min, 12 sec



17

^1H - ^1H Correlations via
COSY and J -coupling
analysis



SL-6-085-crude-500-H

Sample Name:

SL-6-085-crude-500-H

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmrsys/data

Sample directory:

SL-6-085-crude-500-H

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Nov 9 2010

Temp. 25.0 C / 298.1 K

Sample #47, Operator: slevin

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

320 repetitions

OBSERVE C13, 125.6604283 MHz

DECOUPLE H1, 499.7445450 MHz

Power 39 dB

continuously on

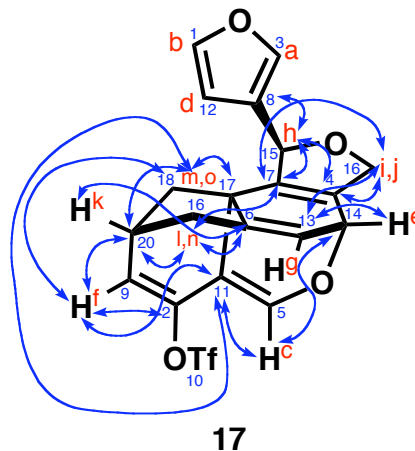
WALTZ-16 modulated

DATA PROCESSING

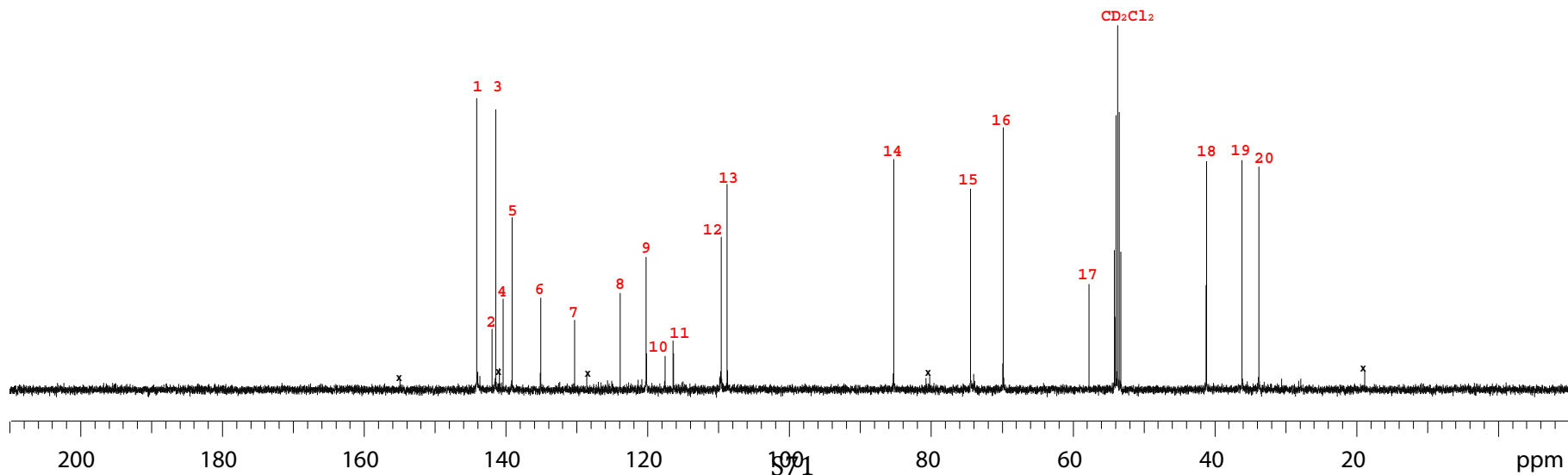
Line broadening 0.5 Hz

FT size 65536

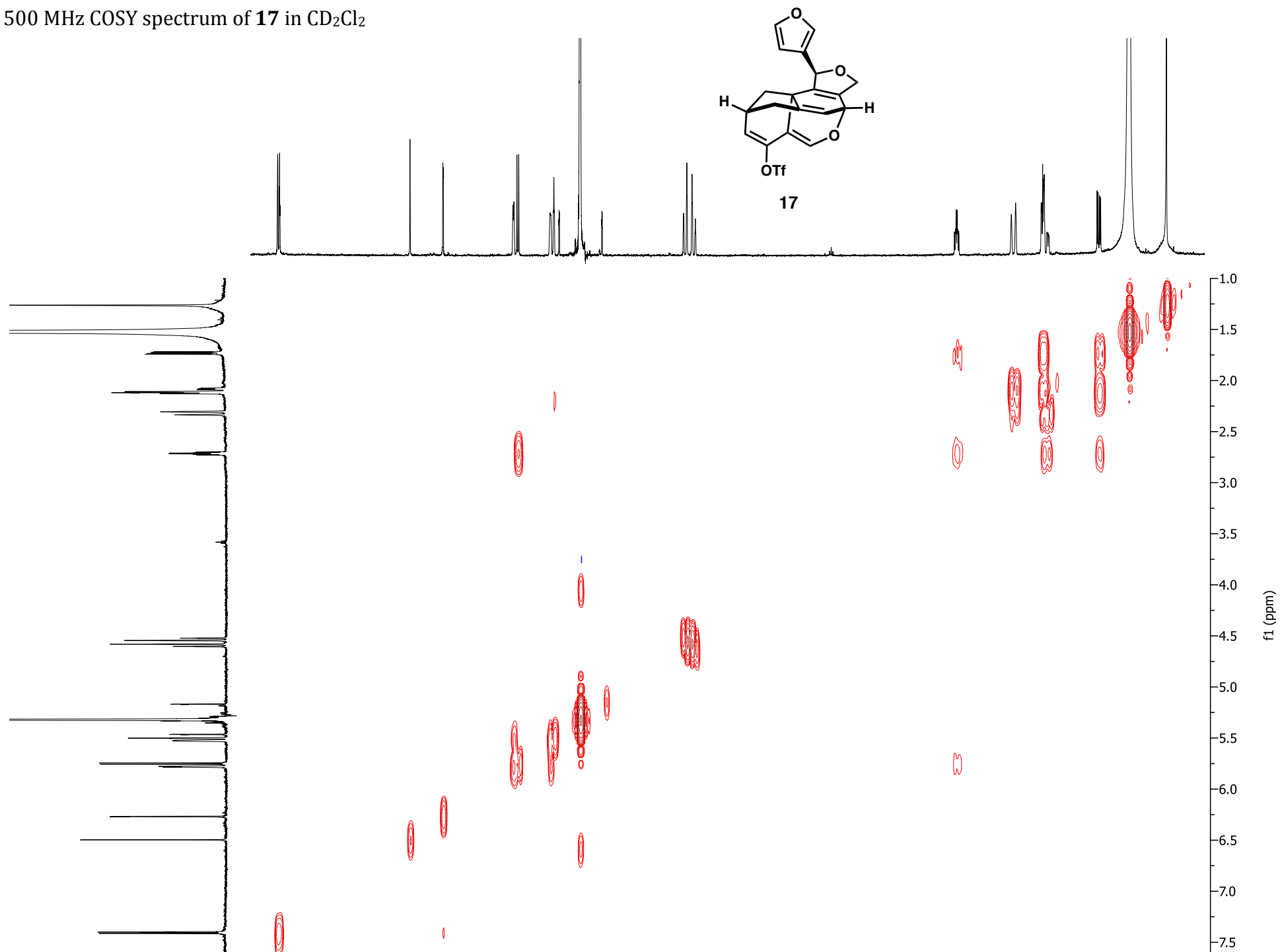
Total time 1 hr, 8 min



HMBC correlations
Carbons are assigned based on
HSQC, HMBC and COSY

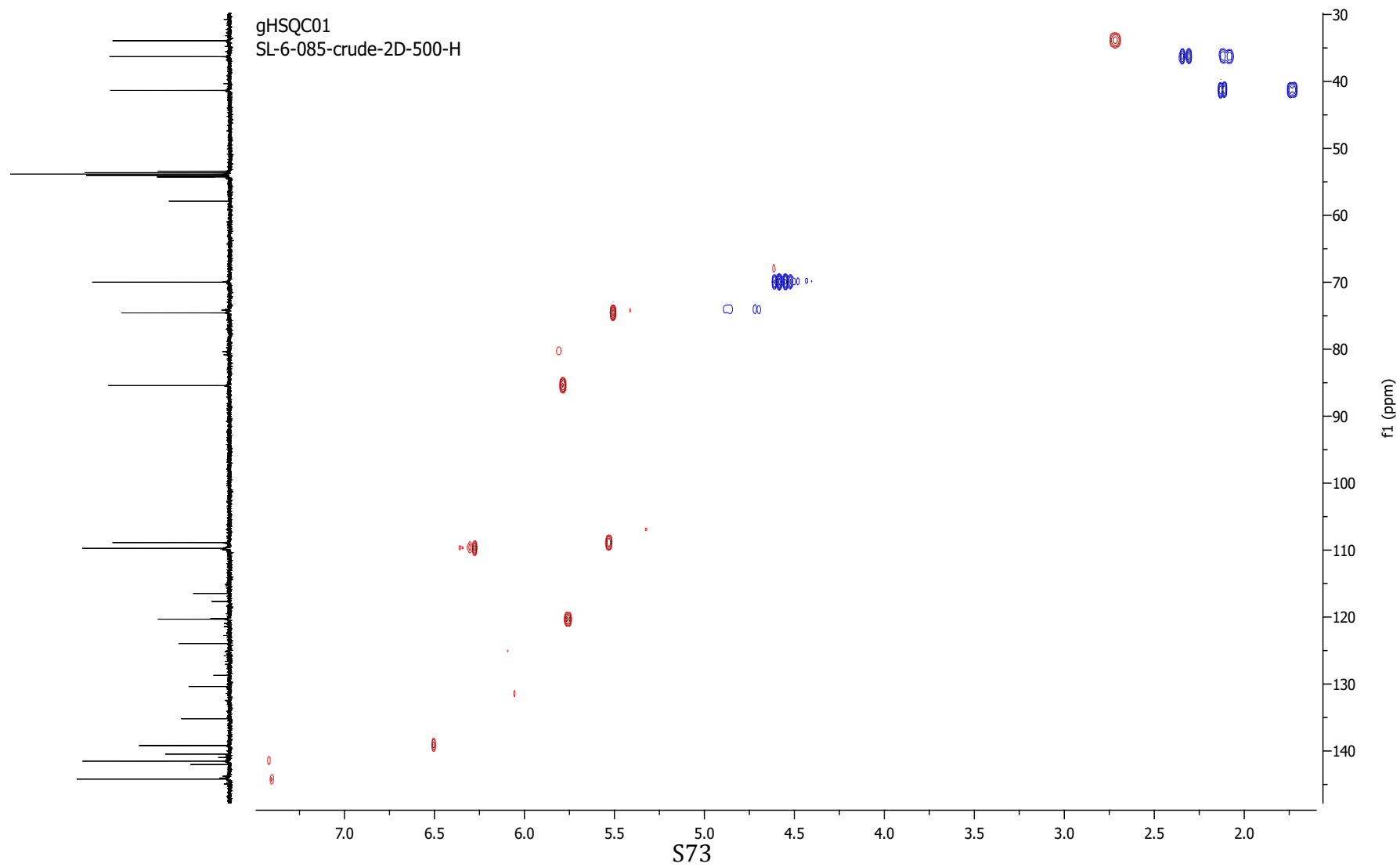
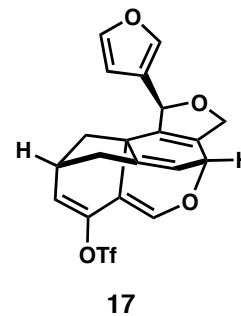


500 MHz COSY spectrum of **17** in CD₂Cl₂



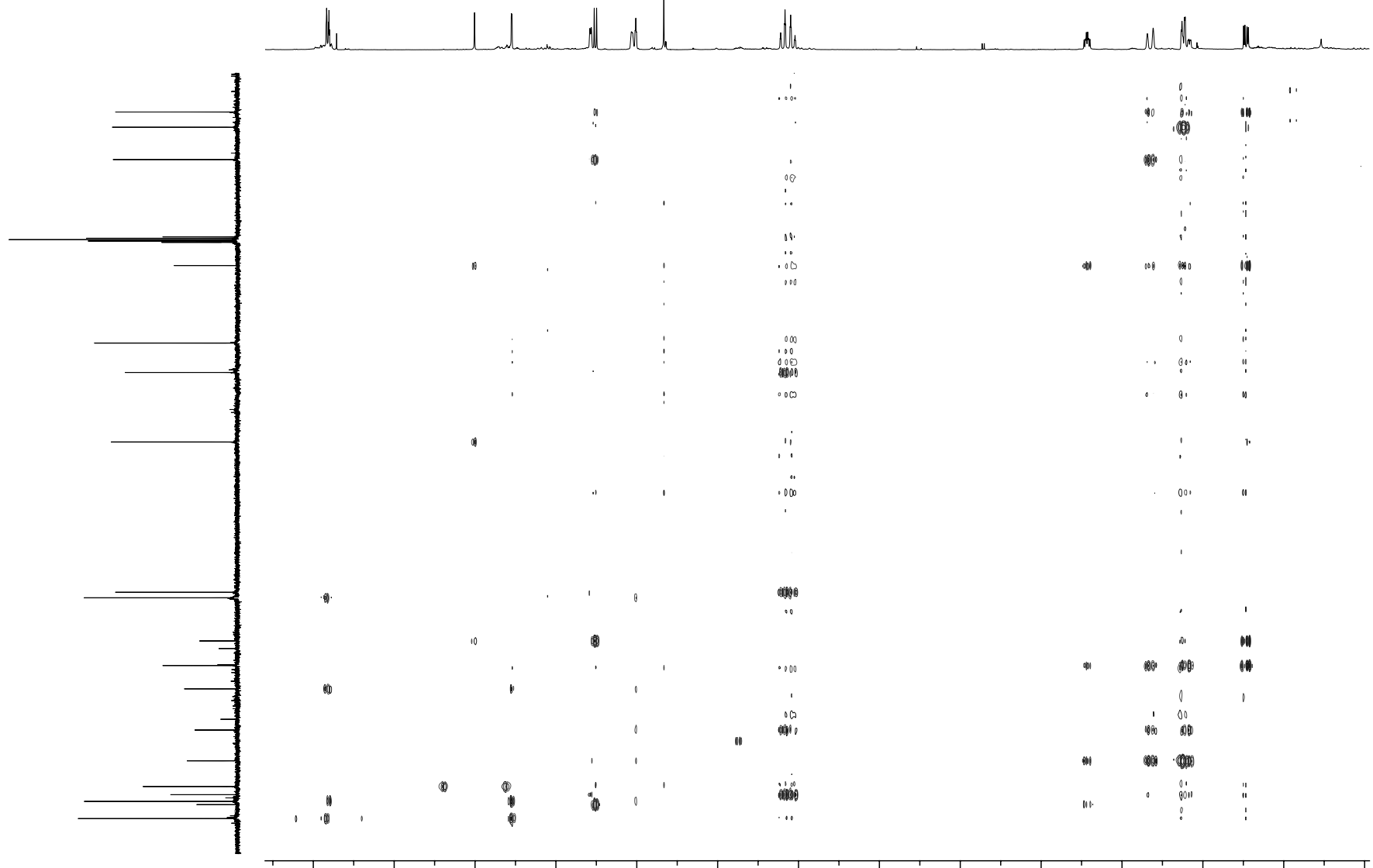
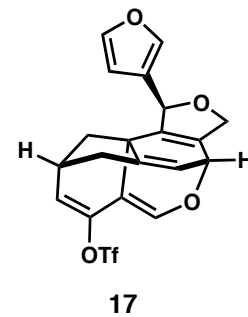
500 MHz HSQC spectrum of unpurified **17** in CD₂Cl₂

X residual CH₂Cl₂



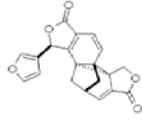
500 MHz HMBC (optimized for 8 Hz coupling)
spectrum of unpurified **17** in CD₂Cl₂

X residual CH₂Cl₂

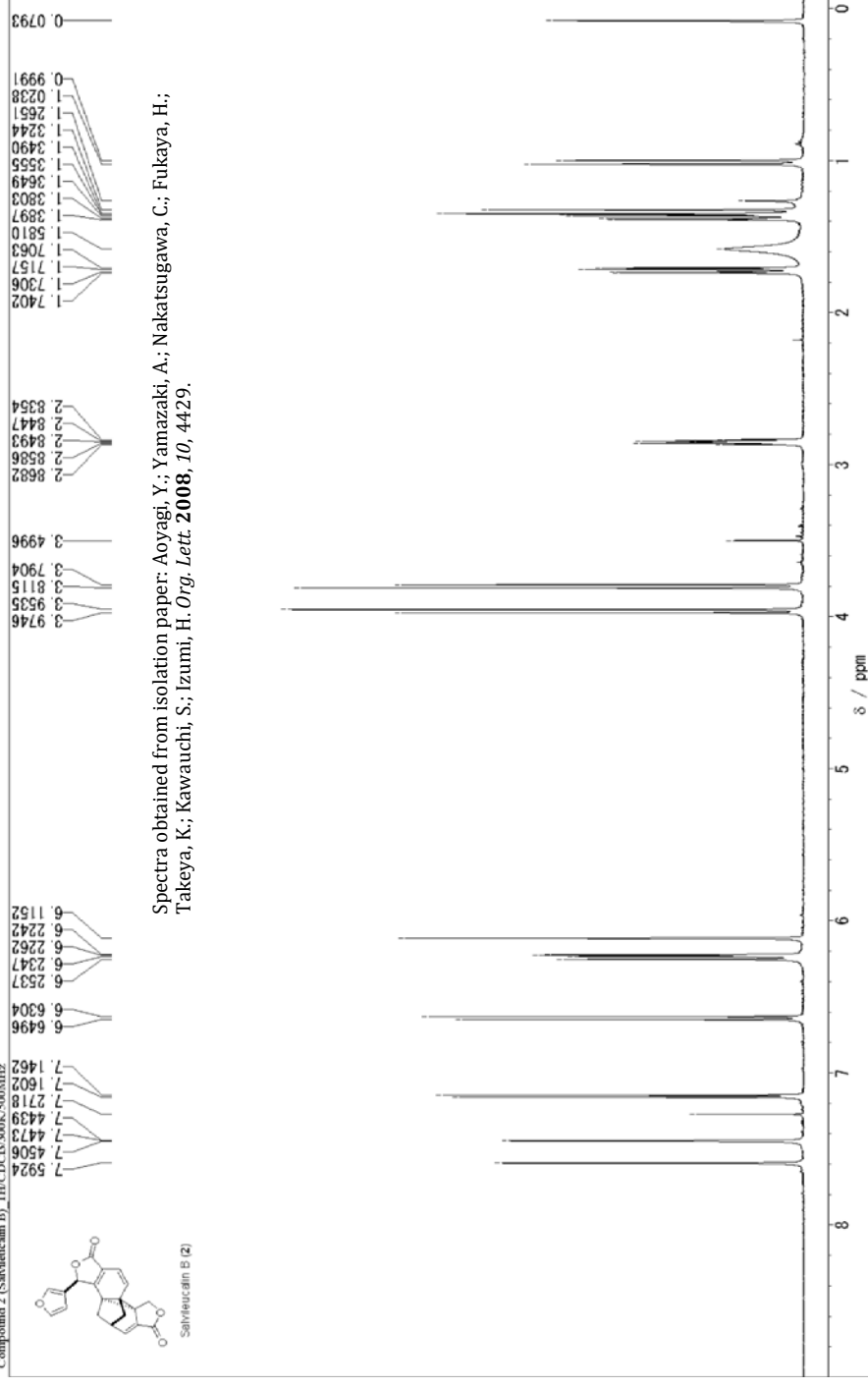


500 MHz ¹H NMR Spectrum of Natural Salvileucalin B in CDCl₃

Compound 2 (Salvileucalin B), 1H/CDCl₃, 300K, 500MHz



Spectra obtained from isolation paper: Aoyagi, Y.; Yamazaki, A.; Nakatsugawa, C.; Fukaya, H.; Takeya, K.; Kawauchi, S.; Izumi, H. *Org. Lett.* **2008**, *10*, 4429.



500 MHz ¹H NMR Spectrum of Synthetic Salvileucalin B in CDCl₃

SL-6-054-column-500-H

Sample Name:

SL-6-054-column-500-H

Data Collected on:

Indy.caltech.edu-inova500

Archive directory:

/home/slevin/vnmr/sys/data

Sample directory:

SL-6-054-column-500-H

FidFile: current

Pulse Sequence: PROTON (c2pul)

Solvent: cdcl3

Data collected on: Oct 24, 2010

Temp: 25.0 C / 298.1 K

Sample #46, Operator: slevin

Relax. delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 2.500 sec

Width: 8000.0 Hz

64 repetitions

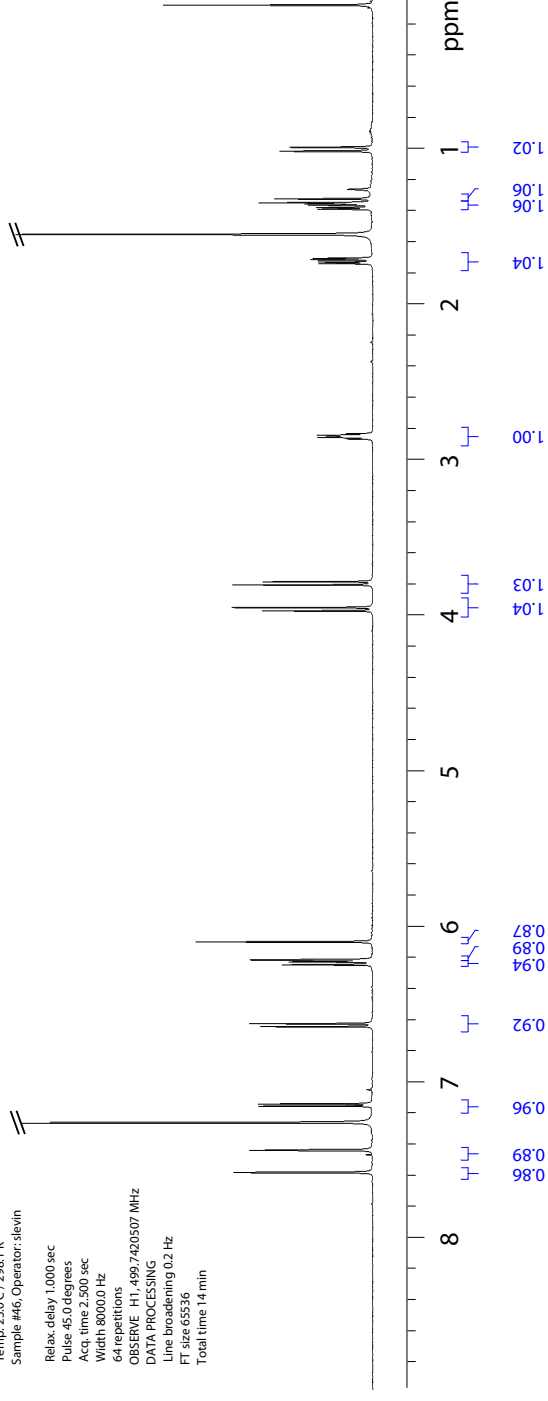
OBSERVE: F1, 499.7420507 MHz

DATA PROCESSING

Line broadening: 0.2 Hz

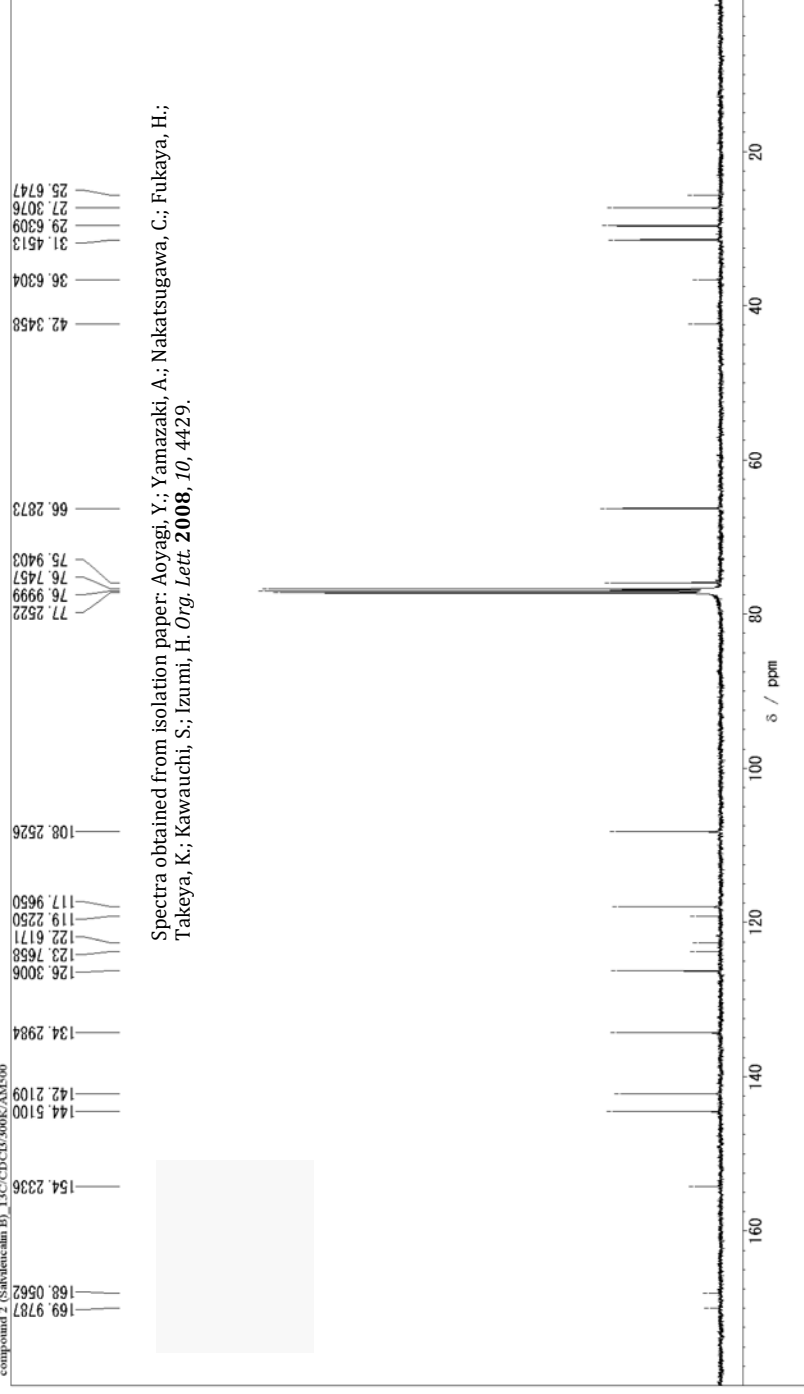
FT size: 65536

Total time: 14 min



125 MHz ¹³C NMR Spectrum of Natural Salvileucalin B in CDCl₃

compound 2 (Salvileucalin B), ¹³C/CDCl₃/300K/AM500



Spectra obtained from isolation paper: Aoyagi, Y.; Yamazaki, A.; Nakatsugawa, C.; Fukaya, H.; Takeya, K.; Kawauchi, S.; Izumi, H. *Org. Lett.* **2008**, *10*, 4429.

125 MHz ¹³C NMR Spectrum of Synthetic Salvileucalin B in CDCl₃

SL-6-067-column-125-13C

Sample Name: SL-6-067-column-125-13C
Data Collected on: indy.cnltech.edu-inova500
Archive directory: /home/slevin/vnmrsys/data
Sample directory: SL-6-067-column-125-13C
FidFile: CARBON01

Pulse Sequence: CARBON (g2pul)
Solvent: cdcl3
Data collected on: Nov 1 2010

Temp, 25.0 C / 298.1 K
Sample #46, Operator: slevin
Relax, delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13500 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 41 min

